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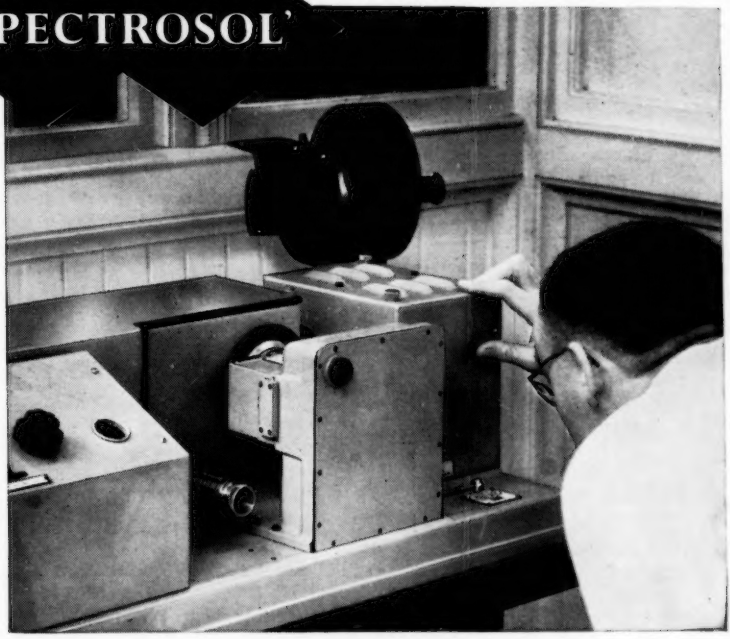

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ANALYTICAL ABSTRACTS

1.—GENERAL ANALYTICAL CHEMISTRY

1594. **Research on the use of organic reagents in analysis.** L. M. Kul'berg, A. G. Kovaleva, A. I. Cherkesov and L. A. Molot. *Uchenye Zapski Saratov. Univ.*, 1954, (34), 126–141; *Referativnŷi Zh., Khim.*, 1955, (9), Abstr. No. 16,515.—The use of alkaloids, quinoline or pyridine in spot tests for the detection of bismuth is discussed. Filter-paper soaked in a chloroform soln. of zinc pyridine thiocyanate and dried gives a red or orange-red spot with $<0{\cdot}02$ μg of Bi in a dilution of <1 in 100,000. The addition of $Pb(NO_3)_2$ soln. further increases the sensitivity, but many ions interfere. Traces of Mo in tungstates or vanadates may be detected by treating the powdered sample with 2 to 3 drops of freshly prepared soln. ($\simeq 5$ per cent.) of ethylenebisdithiocarbamic acid in 15 per cent. acetic acid. In the presence of Mo, an immediate lilac-pink colour appears, changing to brown and then green. In this way $<0{\cdot}01$ per cent. of Mo in vanadate and 0·002 per cent. in tungstate can be detected. Hexanitrohydrazobenzene is recommended as an acid-base indicator (pH range 5·02 to 6·9; colour change pink to violet) especially in the presence of organic solvents. C. D. KOPKIN

1595. **The application of *m*-methoxy-*o*-nitrosophenol to colorimetry.** Tetsuya Torii and Fujiro Iimura (Fac. Sci., Tokyo Univ., Hongo, Tokyo). *J. Chem. Soc. Japan, Pure Chem. Sect.*, 1955, **76** (7), 825–830.—Various reagents for the determination of Co were compared for chelate formation. The cobalt chelate of *m*-methoxy-*o*-nitrosophenol (**I**) has the formula Co.3**I** and is diamagnetic. The bathochromic effect resulting from the chelate formation appears to be due to the marked extent to which the electronegative character of the nitroso group is reduced by the introduction of a methoxy group into the *para* position.

K. SAITO

1596. **Chloramine B as a bromimetric reagent. II. Indirect determinations.** R. C. Paul and Apar Singh (Punjab Univ. Coll., Hoshiarpur, India). *J. Indian Chem. Soc.*, 1955, **32** (10), 694–696.—The indirect determination of $KMnO_4$, $K_2Cr_2O_7$, K_2CrO_4, $KClO_3$, $KBrO_3$, MnO_2 and PbO_2 was carried out with standard chloramine-B reagent; $KClO_3$ in the presence of $KClO_4$, and NaClO in the presence of $KClO_3$, were also estimated by a similar procedure. The weighed sample is treated with a known vol. of standard As_2O_3 solution and 0·5 to 10 g of KBr and then acidified with HCl. The Br liberated is absorbed by As_2O_3 and the excess of the As_2O_3 is determined by titration with chloramine-B solution.

S.C.I. ABSTR.

1597. **8-Hydroxy-2-phenylquinoline and its chelates. I. Preparation and properties of 8-hydroxy-2-phenylquinoline.** G. Bocquet and R. A. Pâris (Fac. des Sci. de Lyon, France). *Anal. Chim. Acta*, 1955, **13** (6), 508–512.—The prep. and properties of 8-hydroxy-2-phenylquinoline are described. The u.v. absorption characteristics in *n*-hexane and in aq. ethanol are determined, and the solubility of the compound in aq. ethanol is measured with the aid of the absorptiometric data. The u.v. spectral absorption curves for aq. ethanolic solutions at various pH values show a number of isosbestic points, the significance of which is discussed.

W. C. JOHNSON

1598. **pH Values of the Clark and Lubs buffer solutions at 25° C.** V. E. Bower and R. G. Bates. *J. Res. Nat. Bur. Stand.*, 1955, **55** (4), 197–200.—The pH values of the Clark and Lubs buffer solutions have been determined at 25° C and the compositions and buffer values are listed at intervals of 0·1 pH unit. The estimated accuracy is $\pm 0{\cdot}02$ pH unit.

O. M. WHITTON

1599. **The application of thio-salts in analysis. IV. A new scheme of qualitative analysis. Part A.** G. B. S. Salaria (Mahendra College, Patiala, India). *Anal. Chim. Acta*, 1955, **13** (6), 513–517.—The common metals are separated into groups, mainly by the formation of hydroxides, sulphides and thio-salts, with the use of *N* Na_2S. The subsequent separation and identification of individual metals follows more conventional lines.

W. C. JOHNSON

1600. **Some observations on the precipitation of group-2 metals in qualitative analysis.** R. Belcher, J. P. G. Farr and J. E. B. Randles (Birmingham Univ., England). *Anal. Chim. Acta*, 1955, **13** (6), 518–525.—After the separation of group-1 metals the filtrate is warmed with 2 ml of aq. H_2O_2 (20 vol.), which is preferred to NH_4I, Hg and other reagents for the reduction of MnO_4' and Cr_2O_7''. Arsenic pentasulphide is pptd. from 6 *N* HCl, and the other sulphides of group 2 from 0·5 *N* HCl, by the method of Holness ("A Concise Scheme of Inorganic Qualitative Analysis," Sir Isaac Pitman & Sons, Ltd., London, 1951). Group-2A sulphides are separated from group-2B sulphides by the use of a soln. of LiOH (1 per cent.) and KNO_3 (5 per cent.) (*cf. Brit. Abstr. C*, 1950, 470). The hot soln. of group-2B sulphides is poured into twice its vol. of conc. HCl, a little H_2S is passed and arsenic sulphides are filtered off. The filtrate is cooled, diluted with an equal vol. of water, H_2S is passed and Sb_2S_3 is filtered off. The filtrate is treated with an equal vol. of 2 *N* aq. NH_3, H_2S is passed and SnS_2 is pptd.

W. C. JOHNSON

1601. **[Identification of] insoluble substances in qualitative analysis.** I. K. Taimni and G. B. S. Salaria (Univ., Allahabad, India). *Anal. Chim. Acta*, 1956, **14** (1), 4–10.—Based on a systematic study of the compounds themselves, a scheme of qual. analysis is proposed for the identification of

the constituents of insol. residues, *e.g.*, after initial decomposition of a sample by fusion or dissolution or both. The following insol. compounds are likely to be present—$BaSO_4$, $SrSO_4$, anhyd. $Cr_2(SO_4)_3$, $PbSO_4$, AgCl, $CrCl_3$, CaF_2, ThF_4, $Zr(HPO_4)_2$, $Ti(HPO_4)_2$, $Ce(HPO_4)_2$, $Th(HPO_4)_2$, $Sn_3(PO_4)_4$, Prussian blue, Turnbull's blue, cupric ferro- and ferri-cyanides, uranyl ferrocyanide, Al_2O_3, Cr_2O_3, SnO_2, Sb_2O_4, SiO_2, ZrO_2, TiO_2, ThO_2, CeO_2, WO_3 and MoO_3. W. J. BAKER

1602. **Standardisation of normal solutions of acids. II. Use of sodium bicarbonate as reference standard.** A. Desjobert and F. Petek (Faculté de Pharmacie, Paris, France). *Anal. Chim. Acta*, 1956, **14** (1), 19–23.—It is inadvisable to use ignited $NaHCO_3$ as working standard in acidimetry because variable titration values are obtained according to the temp. of heating (200° to 700° C) and, to a much less extent, the period of heating (2 to 20 hr.). The product is not identical with that obtained by heating Na_2CO_3 over the same range of temp. and time. An error of 0·26 per cent. can arise in this way when standardising *N* HCl. It is recommended that pure commercial $KHCO_3$, ignited at 250° to 350° C for 2 hr., be used instead of $NaHCO_3$ (*cf. Anal. Abstr.*, 1954, **1**, 875). W. J. BAKER

1603. **Limits of pH governing the use of "amphi-indicators" in two-phase acidimetry and alkalimetry.** G. N. Thomis and A. Z. Kotionis (Manuf. de Mat. Col. d'Athènes, Greece). *Anal. Chim. Acta*, 1956, **14** (1), 11–18.—The use of "amphi-indicators" of the type $B\Delta$ (B = aq. 10^{-2} *M* alkaloid, *e.g.*, atropine; Δ = aq. 10^{-3} *M* indicator, *e.g.*, methyl red) for the titration of organic acids or bases in a mixture (1 + 1, by vol.) of H_2O and $CHCl_3$ is discussed theoretically. The migration of $B\Delta$ from one solvent to the other over a definite range of pH indicates the end-point, but only those amphi-indicators sensitive over a narrow range of pH are of value for amphimetric titrations. Twenty indicators satisfying these conditions and covering the range of pH from 3·5 to 10·3 are listed. For a sharp end-point the final concn. of Δ should be $\simeq 0{\cdot}5 \times 10^{-5}$ *M* and of B $\simeq 10^{-4}$ *M*, with a mol. ratio $B/\Delta = 20$. Applications and advantages of amphi-indicators in the titration of free organic acids and bases under the above-mentioned conditions are discussed. (*Cf. Praktika Akad. Athénon*, 1950, **25**, 182.) W. J. BAKER

1604. **Titrations with extractive end-points. III. The iodine titration.** N. A. Gibson and R. A. White (Univ. Sydney, N.S.W., Australia). *Anal. Chim. Acta*, 1955, **13** (6), 546–549.—Triphenylmethylarsonium iodide (0·5 ml of a 1 per cent. aq. soln.) is used as indicator in the titration of AsO_3''' and S_2O_3'' with 0·1 *N* to 0·01 *N* I, and in reverse titrations. The end-point is marked by the formation (or decomp.) of triphenylmethylarsonium tri-iodide, which imparts a yellow-brown colour to 5 ml of $CHCl_3$ or CCl_4 with which the soln. is shaken. The ions of Cr^{III}, Ni^{II}, Co^{II} and Mn^{II} do not interfere in the titration of S_2O_3''; all but Cr''' interfere in the titration of AsO_3'''. The method cannot be used in the iodimetric determination of Cu'' because the pptd. CuI falls into the organic layer.
W. C. JOHNSON

1605. **The use of decinormal sodium hypochlorite and thiosulphate solutions in oxidimetric determinations in alkaline medium.** J. Bitskei (Polytech. Univ., Budapest). *Magyar Kém. Foly.*, 1955, **61** (12), 406–410.—The simultaneous application of $Na_2S_2O_3$ and NaOCl is based on the reaction—

$$S_2O_3'' + 4ClO' + H_2O = 2SO_4'' + 4Cl' + 2H^{\cdot}.$$

The 0·1 *N* NaOCl is prepared by passing Cl into $\simeq N$ NaOH; *N* NaOH stabilises the soln., which must be kept in the dark. It is standardised in acid medium by iodimetric titration, or with 0·1 *N* $Na_2S_2O_3$. To 0·1 *N* $Na_2S_2O_3$ (20 ml) add 30 per cent. NaOH (5 ml), followed by 1 drop each of 1 per cent. ethanolic brazilin and 5 per cent. KI. Dilute the soln. to 50 ml and, with constant swirling, titrate with 0·1 *N* NaOCl. The colour change is from pale red to yellowish green. For the determination of reducing agents, *e.g.*, sulphites, cyanides, thiocyanates, or of H_2O_2 in strongly alkaline soln., excess of 0·1 *N* NaOCl is added, then a vol. of 0·1 *N* $Na_2S_2O_3$ equivalent to the NaOCl. The excess of $Na_2S_2O_3$ is then back-titrated with 0·1 *N* NaOCl, with brazilin as indicator.
A. G. PETO

1606. **Influence of dissolved oxygen from the air on the results of amperometric titrations of certain anions with lead nitrate.** V. A. Khadeev. *Trudy Sredneaziat. Univ., No.* 55, *Khim. Nauk*, 1954, (7), 25–39; *Referativnŷĭ Zh., Khim.*, 1955, Abstr. No. 40,342.—The presence of dissolved O gives high results in the amperometric titration, with lead nitrate solution, of non-reducible anions, sulphate, oxalate and fluoride. These are caused by a reduction in the total polarographic wave of Pb'' and dissolved O as a result of combination of part of the Pb'' diffusing to the cathode with OH' formed by the simultaneous reduction of O. In the presence of 0·1 to 2·0 ml of 0·01 *N* HNO_3 per 50 ml of solution the positive error is reduced to zero.
G. S. SMITH

1607. **Stability in alkaline solution of dyes used in paper electrophoresis.** C. Moncke (Med. Klin., Rostock Univ., Germany). *Klin. Wochschr.*, 1956, **34** (3–4), 100–101.—A no. of dyes commonly used in Germany for staining protein fractions separated by paper electrophoresis have been examined for their behaviour on extraction with alkaline soln. With the exception of bromophenol blue (**I**), all the dyes tested were readily extractable by means of 0·01 *N* NaOH, whereas for **I**, 5 per cent. Na_2CO_3 was required. For the tests the dyes, dissolved in methanol containing acetic acid, were applied to Whatman No. 1 paper, and dried for 48 hr. at room temp. Elution with alkali for up to 10 hr. had not appreciably affected the extinction values as determined by the step photometer and Lange colorimeter. Amido black 10B was readily destroyed by 0·01 *N* NaOH, but destruction is inhibited by protective colloids, especially by α-globulins.
P. HAAS

1608. **Chemiluminescence method for quantitative analysis.** A. A. Ponomarenko (Lvov Inst. Trade Econ., Ukrain. SSR. *Doklady Akad. Nauk SSSR*, 1955, **102** (3), 539–542.—When solutions of luminol (alkaline solutions of 3-aminophthalhydrazide), oxidant (H_2O_2, hypochlorite) and catalyst (haemin, cuprammonium salts, ferricyanides, other complex salts) are mixed in optimum proportions, a maximum value is obtained for the intensity of luminescence (I); if lower concn. of any single component are taken, a value of $I < I_{max}$ is found. Empirical curves connecting I with concn. of components are constructed for the systems luminol - H_2O_2 or

NaOCl - cuprammonium salts, and these curves serve for determining the concn. of systems containing 0·17 to 20 mg of H_2O_2, 0·012 to 0·5 mg of Cu, or 0·74 to 29 mg of NaOCl, in 20 ml of solution. The concn. of substances that reduce luminescence by reacting with one of the components, usually the oxidant, can be determined similarly, by using empirical curves; examples are aniline (0·0028 to 0·08 mg, reading I 1 min. after mixing) and acetaldehyde (2·5 to 12·5 mg). Addition of glucose to the systems enhances I, and this effect may similarly be applied to the determination of 0·09 to 4 mg of glucose. The differences between results obtained by this and by standard methods do not exceed ±3 per cent. R. TRUSCOE

1609. **Study of the width of spectrographic lines in the arc discharge.** A. Ya. Vyatskin and I. M. Nagibina. *Doklady Akad. Nauk SSSR*, 1954, **98** (1), 59–62; *Referativnyĭ Zh., Khim.*, 1955, Abstr. No. 40,345.—Over certain concn. ranges the width of the lines Mn II 2949, Pb I 2873 and Cd I 3261 A obtained from an a.c. arc discharge is linearly related to the concn. of these elements in the arc. Widening begins at concn. corresponding to bends in the calibration curves. A new method of quant. spectrographic analysis by line-width measurements is proposed. G. S. SMITH

1610. **Some statistical aspects of X-ray fluorescence spectrometry.** W. S. Horton (Knolls Atomic Power Lab., General Electric Co., Schenectady, New York, U.S.A.). *Applied Spectroscopy*, 1955, **9** (4), 173–177.—The precision of X-ray fluorescence spectrometry can be deduced by the application of a simple derivation of the probability distribution function to the estimation of the time required to accumulate a preset number of counts. Good agreement is shown between the theoretical and practical precision attained for the estimation of Ta in steel. P. T. BEALE

1611. **Analysis and preparation of stable isotopes.** M. Roth (Commiss. Energie Atomique, France). *Chim. Anal.*, 1956, **38** (1), 3–14.—In this review the analysis of isotope mixtures by the mass spectrometer is described and orders of magnitude of resolution, sensitivity and stability of supplies are given, with examples applied to particular elements. Solid elements may be vaporised in the ion chamber by heating or by utilising thermo-ionic emission from the cathode on which the element is placed, usually as the nitrate. Microgram quantities only are required for analysis. Ion currents of about 10^{-17} amp. may be measured by sensitive electron multipliers. An instrument of high efficiency and dispersion detects concn. of 1 p.p.m. Other physical and chemical methods of isotope separation are tabulated, with range of application, energy consumed per gram of isotope separated and magnitude of quantities produced. The preparation of heavy water by electrolysis and by water-hydrogen exchange reaction is described. G. A. BASSETT

See also Abstracts 1612, 1613, 1614, 1615.

2.—INORGANIC ANALYSIS

1612. **Use of azo dyes as reagents in inorganic analysis and as antibacterials. I.** R. Das and S. S. Guha Sircar (Ravenshaw College, Cuttack-3, India). *J. Indian Chem. Soc.*, 1955, **32** (10), 679–686.—Fifteen azo compounds were prepared by coupling salicylic acid with diazotised *o*- and *m*-nitroanilines, sulphanilamide, *p*-aminoacetophenone and *o*-amino-benzoic acid, and 8-hydroxyquinoline with the same compounds and also with *p*-nitroaniline, *m*-toluidine, *m*- and *p*-aminobenzoic acids, and *p*-aminosalicylic acid. They were then examined systematically as reagents for spot tests and for the gravimetric and colorimetric determination of some 20 metals. Quant. estimations of Pb and Zn were carried out with the coupled azo compounds from salicylic acid and *o*-nitroaniline, salicylic acid and *o*-aminobenzoic acid (for Pb only) and 8-hydroxyquinoline and *p*-aminobenzoic acid (for Pb and Zn). S.C.I. ABSTR.

1613. **Precipitation of metals with aminophenylarsonic acids.** R. Pietsch (Univ. Graz, Austria). *Mikrochim. Acta*, 1955, (5–6), 954–961.—The precipitation of a large number of metal ions with *o*-, *m*- and *p*-aminophenylarsonic acids has been investigated. Many are shown to be precipitated quantitatively by one or other of these acids. C. A. SLATER

1614. **Precipitation of metals with nitrophenylarsonic acids.** R. Pietsch (Univ. Graz, Austria). *Mikrochim. Acta*, 1955, (5–6), 1019–1025.—The precipitation of a large number of metal ions with *o*-, *m*- and *p*-nitrophenylarsonic acids has been investigated. In many instances the pptn. is quantitative. C. A. SLATER

1615. **The use of disubstituted dithiocarbamates in micro-analysis. IV.** H. Malissa and E. Schöffmann (Max-Planck-Inst., Düsseldorf). *Mikrochim. Acta*, 1955, (5–6), 1057–1060.—The use of dithiocarbamates for gravimetric micro-determinations of silver, nickel, cobalt, iron and copper is described. C. A. SLATER

1616. **Co-precipitation of certain ions with ferric hydroxide in ammoniacal chloride medium.** D. P. Shcherbov and I. I. Sagalovich. *Izvest. Akad. Nauk Kazakh. SSR, Ser. Khim.*, 1955, (8), 114–117; *Referativnyĭ Zh., Khim.*, 1955, Abstr. No. 40,324.—The order of the degree of co-pptn. of certain metal ions with $Fe(OH)_3$ from solutions of the ammonia complexes is $Co^{++} > Ni^{++} > Zn^{++} > Cu^{++} > Cd^{++} > Co^{+++} > Ag^{+}$. When the ionic radii are similar, co-pptn. increases with increase in the instability of the ammonia complex. The behaviour of the ammonia complexes of Cu, Zn and Cd in the presence of Fe are studied in greater detail since these metals in polymetallic ores are frequently determined polarographically in an ammoniacal chloride supporting electrolyte. In a solution 0·7 N in NH_4Cl and 3·0 N in NH_3 and also in a solution 1·5 N in NH_4Cl and 5·0 N in NH_3 hardly any co-pptn. of Cd^{++} with $Fe(OH)_3$ occurs. Co-pptn. of Cu^{++} starts only with >120 mg of Fe^{+++} (vol. not given). With low concn. of the supporting electrolyte, Zn^{++} begin to ppt. when 40 mg of Fe^{+++} are present, but with high concn. pptn. does not occur until ⩾80 mg of Fe^{+++} are present. In polarographic determinations of Zn in polymetallic ores it is necessary to increase the amount of ammonia in the solution when the content of Fe is high or variable. Standard solutions for the construction of calibration curves should have iron contents corresponding to those of the samples being analysed. G. S. SMITH

1617. **Analytical applications of the demasking of ferrocyanide.** F. Feigl and A. Caldas (Min. Agric., Rio de Janeiro, Brazil). *Anal. Chim. Acta*, 1955, **13** (6), 526–530.—Ferrocyanides are decomposed by substances that form stable cyanides, and the resulting Fe^{++} are identified with 2:2′-dipyridyl. Thus $Fe(CN)_6''''$ and the salts of Hg, Ag and Pd can be detected. *Detection of ferrocyanide*—To 1 drop of the acid, neutral or alkaline (with Na_2CO_3) soln. in a micro test-tube add 1 drop of 1 per cent. alcoholic 2:2′-dipyridyl soln. and a few mg of $HgNH_2Cl$ and warm the soln. in a water bath. Acid soln. should be set aside for a few min. before being warmed. A red colour is produced by 0·2 μg of $K_4Fe(CN)_6$ even in the presence of 1 mg of $K_3Fe(CN)_6$. *Detection of the salts of Hg, Ag and Pd*—To 1 drop of 1 per cent. $K_4Fe(CN)_6$ soln. add 1 drop of 2 *N* aq. NH_3, 1 drop of 1 per cent. alcoholic 2:2′-dipyridyl soln. and 1 drop of the test soln. and warm in a water bath. A red colour is obtained with 0·5 μg of Hg, 2 μg of Ag or 2 μg of Pd. A similar reaction is obtained with the halides of Ag and with AgCN, AgSCN, $Ag_4Fe(CN)_6$, $Ag_3Fe(CN)_6$ and $Ag_3Co(CN)_6$ when these substances are added in the solid state. W. C. JOHNSON

1618. **Inorganic paper-chromatography with tetrahydrofuran.** H. Hartkamp and H. Specker (Inst. Spektrochem. u. Angew. Spektroskop., Dortmund-Aplerbeck, Germany). *Naturwissenschaften*, 1955, **42** (19), 534–535.—The developing solvent is a mixture of tetrahydrofuran and conc. HCl (50:15 by vol.). The ions Fe^{+++}, MoO_4'', Zn^{++} and Cd^{++} run with the solvent front, but UO_2^{++}, Cu^{++}, Co^{++}, VO_2^{+}, Mn^{++} and Ni^{++} separate well; the velocities are in the order given. E. KAWERAU

1619. **Concentration of inorganic ions on paper by means of electrophoresis.** G. de Vries and E. van Dalen (Freien Univ., Amsterdam). *Anal. Chim. Acta*, 1955, **13** (6), 554–561.—For spot tests, various metal ions and some anions are concentrated into a smaller area by applying a current from the tip of a platinum wire. By this means the limiting sensitivity of such tests is increased to between 1 in 10^6 and 1 in 10^7. The spots produced in paper chromatograms are concentrated similarly so that, *e.g.*, 0·001 μg of Ni^{++}, 0·002 μg of Co^{++} and 0·003 μg of Mn^{++}, originally present in 25 μl of soln., can be detected, and any pair of these metals in quantities of 0·01 μg can be detected in the presence of 500 μg of the third (except Mn^{++} in the presence of Co^{++}). If the concentration precedes the chromatographic separation, separation can be completed in a few min. W. C. JOHNSON

1620. **The application of spectrographic solution and powder methods to the analysis of miscellaneous samples.** P. T. Beale and D. A. Poynter (British Non-Ferrous Metals Research Ass., London). *Metallurgia*, 1955, **52**, 253–261.—The various spectrographic solution and powder methods in use in the British Non-Ferrous Metals Research Association laboratory are detailed and the factors affecting the choice of method, such as solubility of sample, elements to be determined, and sensitivity and accuracy required, are discussed. Procedures for conversion of sample to solution or powder and for preparation of standards are given for Al, Cu, Pb and their alloys, and Co, Cr, Ti and Zn. A detailed description is given of the following methods of sparking solutions or powders, including the preparation and mounting of electrodes: (*a*) porous-cup electrode; (*b*) copper-cup; (*c*) powder spark; (*d*) powder arc; (*e*) rotating disc; (*f*) flame; and (*g*) impregnated electrode. Data are presented to show the wide applicability of spectrographic analysis and the accuracy expected and, where available, comparisons are made with chemical analysis. G. C. JONES

1621. **Methods for measuring the hydrogen content of weld metal.** P. D. Blake. *Brit. Welding J.*, 1955, **2**, 558–561.—A description is given of the methods used in the laboratories of an electrode-manufacturing company for measuring the diffusible and residual hydrogen contents of ferrous weld metal. These two values can be determined separately, by collecting the diffusible hydrogen under mercury at room temp. and subsequently extracting the residual hydrogen by vacuum heating; alternatively the total hydrogen may be determined in a single step by vacuum heating. G. C. JONES

1622. **Acetylene method for the determination of water.** J. Minczewski and J. Tromszczyński. *Przem. Chem.*, 1955, **11**, 147–149.—Work carried out in this field by various authors is discussed, and the conclusions have been verified. The method is not as general as suggested, for its accuracy must be checked against that of others for a given substance. The volume of acetylene equivalent to 1 g of water must be found empirically for each sample of CaC_2 used. Results depend on the form in which the water is bound to the investigated substance, on its concentration and that of the carbide, the solubility of acetylene in the substance, and the time and temperature of the reaction. A. O. JAKUBOVIC

1623. **Gasometric micro-determination of water in hydrated salts.** A. A. Sirotenko (Columbia Univ., New York). *Mikrochim. Acta*, 1955, (5–6), 917–920.—The method is based on the Elitsur gasometric procedure. The water is extracted from the hydrated salt with anhydrous pyridine, and allowed to interact with calcium hydride to liberate hydrogen. An apparatus is described. The results of the determinations are reproducible to within 0·3 per cent. of theoretical. C. A. SLATER

1624. **Determination of hydrogen peroxide in alkaline solution.** J. Bitskei (Tech. Univ., Budapest). *Acta Chim. Acad. Sci. Hung.*, 1955, **8** (1–3), 203–206.—To determine H_2O_2 in alkaline solution, a known amount of NaOCl is added, then excess of NaOCl is destroyed by adding an excess of As_2O_3, and the excess of As_2O_3 is back-titrated against NaOCl with KI as catalyst and brazilin as indicator. *Procedure*—To the H_2O_2 solution containing 2 *N* NaOH (10 ml) add a known excess of 0·1 *N* NaOCl (20 to 30 ml) and shake the soln. to drive off O. Then add an equiv. amount of 0·1 *N* As_2O_3, $KHCO_3$ (2 g), 5 per cent. KI (1 drop) and 1 per cent. alcoholic brazilin (1 drop), stir the soln. and titrate against 0·1 *N* NaOCl until the colour changes from red to yellow-green. Thus approx. 25 and 50 mg of H_2O_2 can be determined with an accuracy within $\simeq \pm 0{\cdot}4$ and $\pm 0{\cdot}1$ per cent., respectively. The method is generally applicable to peroxides. J. P. STERN

1625. **The flame-photometric determination of the alkali metals.** E. Pungor and E. É. Zapp (Eötvös Loránd Sci. Univ., Budapest). *Magyar Kém. Foly.*, 1955, **61** (12), 421–427.—A Beckmann DU spectrophotometer was used, with a 0·1-mm slit. The

wavelengths studied were Li 6707·8 A, Na 5890 A, K 7664·9 A and 7699 A. The doublets were not resolved. The optimum pressures of H and O were determined for each nozzle. It was found that Li, Na and K do not interfere with each other; the other alkali metals interfere more. In a mixture of Na and K, the mean error of the determination of Na was 1·82 per cent., and of K, 1·14 per cent. After the Lawrence - Smith procedure, the alkali metals must be determined in the presence of much Ca. This can be done in the presence of 10^{-3} ionic wt. per litre of Ca, but if 10^{-1} ionic wt. per litre is present, there is some error, decreasing in the order Li, Na, K. With all these metals, the interference of Ca is additive and can be calculated.

A. G. PETO

1626. **A colorimetric method for the determination of sodium.** P. N. Kovalenko and V. V. Ten'kovtsev. *Ukrain. Khim. Zhur.*, 1954, **20** (4), 411–416; *Referativnyĭ Zh., Khim.*, 1955, (9), Abstr. No. 16,552.—For the determination of small quantities of Na, a soln. of zinc uranyl acetate is added to 1 ml of the solution to be analysed, and the ppt. of $NaZn(UO_2)_3(CH_3COO)_9.6H_2O$ is washed with two 0·5-ml portions of a mixture (9:1) of ethanol and acetic acid, saturated with zinc uranyl acetate (80 per cent.). The ppt. is dissolved in water acidified with H_2SO_4, and NH_4SCN is added to give a final concn. of 1·5 *N*. The soln. is diluted to 10 ml and the absorption is measured after 30 min., with blue filters. It has been established that for 1 mg of Na it is necessary to use $\not<1$ ml of the reagent. The optimum conditions are a reagent vol. 1·5 times the vol. of soln., and a pptn. time of 2 hr. An empirical conversion factor of 0·0339, or a calibration curve, may be used. In both cases the error is $\simeq 0{\cdot}5$ per cent. A study of the electrode polarisation during reduction of U^{VI} on the dropping-mercury electrode in NH_4SCN soln. has shown that the reduction proceeds from U^{VI} to U^{IV}, and that the $UO_2^{\cdot\cdot}$ complex with NH_4SCN is $(NH_4)_2[UO_2(SCN)_4]$.

C. D. KOPKIN

1627. **The determination of sub-microgram quantities of sodium in lithium metal by radio-activation.** A. A. Smales and B. A. Loveridge (Atomic Energy Res. Estab., Harwell, England). *Anal. Chim. Acta*, 1955, **13** (6), 566–573.—The method described is suitable for the determination of contents of Na down to 0·02 p.p.m. in lithium metal. Four samples, in blocks 3 mm × 3 mm × 15 mm, are sealed in silica phials and submitted to neutron activation. After 2 to 3 hr. "cooling," the samples are dissolved in ethanol and dil. HCl with the addition of NaCl to provide the carrier. Ethanol saturated with HCl is added to ppt. NaCl, which is centrifuged, washed with ethanol and redissolved. The Na is then pptd. as the triple acetate of Na, Zn and $UO_2^{\cdot\cdot}$. The triple salt is treated with ethanol saturated with HCl and the pptd. NaCl is washed with ethanol. The cations are then absorbed on a column of Zeo-Karb 225 and eluted with 1·1 *N* HCl, when the $Na^{\cdot}$ are obtained in the first fraction and are thus separated from bi- and ter-valent cations. Ethanolic HCl is added to the Na fraction and the pptd. NaCl is finally submitted to a β-count. Standards are provided by two aliquots (0·1 g) of NaCl soln. (100 p.p.m. of Na) submitted to the same procedure. A correction graph for self-screening effects in the sample is provided.

W. C. JOHNSON

1628. **Flame-photometric determination of sodium, potassium, calcium, magnesium and manganese in glass and raw materials.** N. Roy (Thatcher Glass Mfg Co., Elmira, N.Y., U.S.A.). *Anal. Chem.*, 1956, **28** (1), 34–39.—Soda-lime and boro-silicate glasses and similar materials, such as sand, slag and felspar, are treated with a mixture of HF and $HClO_4$. The resulting salts are re-dissolved in HCl and excited in the hydrogen-flame attachment to the Beckman model B spectrophotometer. A wide range of contents is covered with an accuracy usually within 0·5 per cent. of the content. Various inter-element effects and interferences are noted.

P. T. BEALE

1629. **S-Substituted thioureas as analytical reagents. I. S-Methylthiuronium sulphate as a reagent for copper and nickel.** S. K. Siddhanta and P. C. Kundu (Inst. Tech., Kharagpur, India). *J. Indian Chem. Soc.*, 1955, **32** (10), 655–662.—Nickel and Cu can be quant. pptd. as methylmercaptides in ammoniacal (or for Cu, alkaline tartrate) solution. The ppt. can be dried at 100° C to its stoicheiometric composition in each case. Separation of Cu and Ni, and of Cu and Zn, can also be effected in ammoniacal solution containing sufficient NH_4Cl or ammonium oxalate. In each case, double pptn. is required. Copper methylmercaptide $[Cu(SCH_3)]$ can be weighed as such, and Ni and Zn estimated in the two filtrates by dimethylglyoxime and as pyrophosphate, respectively.

S.C.I. ABSTR.

1630. **Chelatometry. II. Ethylenediaminetetra-acetic acid (EDTA) and the cupric - cuprous redox system.** P. Wehber (Metallhütte Mark A.-G., Hamburg). *Mikrochim. Acta*, 1955, (5–6), 927–933.—Cupric ions can be titrated with 0·01 *M* EDTA in the presence of excess of NH_4SCN and dilute HCl. Variamine blue B is used as a redox indicator. The titration of EDTA with cupric salts is also possible. Less than $\simeq 3$ mg of Cu and 18 mg of EDTA can be determined.

C. A. SLATER

1631. **A new spot-test for silver.** B. Sen and P. W. West (Louisiana State Univ., Baton Rouge, U.S.A.). *Mikrochim. Acta*, 1955, (5–6), 979–982.—Nickel dimethylglyoxime (0·5 g) is suspended in KCN solution (0·4 per cent.) (100 ml) for 24 hr. The solution is filtered and stored in the absence of light. Whatman No. 1 papers are dipped into the solution, rapidly dried in infra-red light, and stored in a stoppered bottle. When treated with a neutral solution containing $Ag^{\cdot}$ the test papers become red; 0·5 μg of Ag at a concentration of 1 in 100,000 can be detected. Gold (tervalent) and nickel (bivalent) interfere.

C. A. SLATER

1632. **Electrometric determination of silver in ammoniacal solution by a method of electrode depolarisation.** E. Michalski and W. Jędrzejewski (Univ. Lodz, Poland). *Roczn. Chem.*, 1955, **29**, 862–870.—Electrometric determinations of Ag, alone and in the presence of $Fe^{\cdot\cdot\cdot}$, $Cu^{\cdot\cdot}$ and $Pb^{\cdot\cdot}$, are carried out with KI solution in aq. NH_3 soln. The accompanying ions are kept in solution as complex tartrates. The closed-circuit electrolytic arrangement consists of a short (3 mm) platinum wire as indicator electrode, a similar platinum wire, immersed in a saturated $Hg_2(NO_3)_2$ solution, as reference electrode, and a spot galvanometer of sensitivity 10^{-8} amp/mm/m, resistance 350 ohms. The test solution is connected with the half-electrodes by an electrolytic bridge filled with

saturated KNO_3 solution. The end-point is established by the depolarisation of the indicator electrode (anode), brought about by the first excess of added I′, when the galvanometer shows an increase of current tension. The linear relation between increase of current and volume of added reagent permits the determination of the end-point by amperometric diagrams. The method yields results with <0·1 per cent. error for determinations of pure silver (28 mg of Ag in 70 ml of aq. NH_3 soln.); in mixtures of Ag and Fe (1:10) or Ag, Cu and Pb (1:10:10) the error is <0·5 per cent.

S.C.I. Abstr.

1633. **The titration of the silver and iodide ions with reversible redox adsorption-indicator. I.** J. Bognár and O. Jellinek (Rákosi Mátyás Tech. Univ. for the Heavy Industries, Miskolc, Hungary). *Magyar Kém. Foly.*, 1955, **61** (12), 410–416.—When I′ are titrated with $AgNO_3$, the dye Xylene blue VS is adsorbed on the surface of the ppt. and is an acid - base indicator. In the presence of iodine it is a redox indicator. *Procedure*—When titrating with a 0·1 *N* $AgNO_3$ soln., 1 drop (≃0·03 ml) of 0·1 *N* KIO_3 is added, and for 50 to 100 ml of unknown soln. 0·2 to 0·3 ml of 0·1 per cent. Xylene blue VS soln. The colour change is yellow to red. Vigorous shaking helps the coagulation of the ppt. When 1 drop of 0·1 *N* ethanolic iodine is used instead of KIO_3, the accuracy decreases slightly. The titration of Ag^+ with 0·1 *N* KI is carried out similarly. Most of the KI soln. should be added quickly and the soln. shaken vigorously before the end-point. The colour change is red to yellow. The accuracy is greater when 1 drop of ethanolic iodine is used instead of 1 drop of 0·1 *N* KIO_3. In both cases the results of the more accurate method agree well with those of potentiometric titrations. The addition of 1 drop of the titrating soln. to 1 litre of 0·0005 *N* unknown soln. still causes a noticeable colour change. Strong acids do not interfere; Cl′ and Br′ do.

A. G. Peto

1634. **Determination of gold in the presence of copper and cadmium.** V. Ivković and S. Saula (Chem. Tech. Inst., Belgrade). *Bull. Soc. Chim. Belgrade*, 1955, **20** (7), 465–474.—Low results are obtained when Au is determined in the presence of Cu or Cd by reduction with alkaline H_2O_2. If HCl or H_2SO_4 is used to dissolve the CuO only 64 to 76 per cent. of Au is recovered, according to the concn. of acid. If 6 per cent. H_2SO_3 is used to dissolve the CuO only 94 per cent. of Au is recovered owing to incomplete coagulation. If HCl is added to the H_2SO_3 as a coagulant slightly high results are obtained owing to occlusion, but the error is then <0·5 per cent. of the amount of Au if the ratio Au to Cu + Cd is ≤1:3.

A. B. Densham

1635. **V. Notes on spectrographic analysis [of alkaline-earth sulphates].** C. O. Harvey. *Bull. Geol. Surv. G.B.*, 1955, (9), 50–55.—A technique for the analysis of natural alkaline-earth sulphates is described. Samples are ignited at 900° C to expel combined water, then finely ground and mixed with three times their wt. of pure carbon powder. The mixture is excited in a crater in the anode of a d.c. carbon arc. Synthetic standards are used and photographed on each plate. Details of exposure conditions and line pairs are given for the determination of $BaSO_4$ in $CaSO_4$ (0·03 to 0·2 per cent.), $SrSO_4$ in $CaSO_4$ (0·1 to 1·0 per cent.), $CaSO_4$ in $BaSO_4$ (0·2 to 4·0 per cent.), $CaSO_4$ in $SrSO_4$ (0·2 to 2·0 per cent.), $BaSO_4$ in $SrSO_4$ (1 to 40 per cent.) and $SrSO_4$ in $BaSO_4$ (0·2 to 80 per cent.). A table shows analyses of 15 minerals with good agreement with chemical determinations where available. Special spring clamps, to fit the Hilger - Gramont arc stand, were designed to hold the carbon electrodes, and a simple semi-mechanical powder-mixing device was constructed.

P. T. Beale

1636. **Colorimetric determination of magnesium with 2-naphthol-3-(2:4-dimethyl)-carboxanilide-1-azo-(2-phenol-5-sulphonic acid).** C. K. Mann (Univ. of Virginia, U.S.A.). *Dissert. Abstr.*, 1955, **15** (10), 1717–1718.—A determination of trace quantities of Mg in the range 0·02 to 0·4 p.p.m. is based on the formation of a coloured complex between Mg^{++} and the sodium salt of 2-naphthol-3-(2:4-dimethyl)-carboxanilide-1-azo-(2-phenol-5-sulphonic acid). This acid - base indicator is blue in the pH range 8 to 11 but changes to red in the presence of Mg. The optimum pH is 8·90 to 8·95, and may be obtained with a borax buffer. Under these conditions the sensitivity is 1 part of Mg in 50 million parts of solution. The standard deviation within the optimum concn. range (0·12 to 0·40 p.p.m.) is ±2·5 to 7·5 per cent. With biological materials and complex organic mixtures, Mg must first be separated from most other bivalent and tervalent metal ions by extracting with 8-hydroxyquinoline and, to remove any remaining Ca, separating on an ion-exchange column. Calcium need not be separated unless it will be present in the final solution to the extent of 12 p.p.m. or more.

O. M. Whitton

1637. **Rapid method of determining the magnesite content of chrome magnesite lining.** S. T. Balyuk. *Ogneuporȳ*, 1954, (2), 84–86; *Referativnȳĭ Zh., Khim.*, 1955, Abstr. No. 21,492.—An average sample is reduced by quartering from 1 kg to 30 to 40 g, dried for 7 min. at 200° to 230° C, crushed while hot, sieved through a mesh of 400 apertures per sq. cm, and then mixed with a small pestle in a porcelain mortar. In a 500-ml flask fitted with an air-condenser 1 g is gently boiled for 8 min. with 50 ml of *N* HCl. The condenser is washed down with water, the contents of the flask are mixed with 200 ml of water and 5 to 10 drops of 1 per cent. phenolphthalein solution, and titrated with *N* NaOH, with vigorous shaking, to a permanent pink coloration. The time for a determination is 40 to 45 min.

G. S. Smith

1638. **The complexiметric determination of calcium in phosphate-containing materials. I. The removal of phosphate with ammonium molybdate.** L. Kálmán and Á. Vágó (Res. Inst. Heavy Chem. Ind., Veszprém, Hungary). *Magyar Kém. Foly.*, 1955, **61** (12), 416–419.—Phosphate is pptd. as molybdophosphate and filtered off; calcium is then determined by titration with EDTA (disodium salt), with murexide as indicator, in the usual way.

A. G. Peto

1639. **Rapid heterometric micro-determination of calcium as sulphate in excess of magnesium [ions] or other supplements.** M. Bobtelsky and J. Eisenstadter (Hebrew Univ., Jerusalem, Israel). *Anal. Chim. Acta*, 1956, **14** (1), 89–95.—A direct heterometric titration of 10 ml of a strongly ethanolic soln. of $CaCl_2$, or $Ca(NO_3)_2$, containing 2 to 4 mg of Ca^{++} in addition to 2 ml of 1 to 2 *M* $MgCl_2$, can be made with 0·04 *M* H_2SO_4 in 90 per cent. v/v ethanol. The titration takes ≃15 min. at 18° C, the end-point being the intersection of the curve of max. optical density with the last part of the titration curve. The accuracy is within 2 per cent.

The presence of other additives besides $MgCl_2$, *e.g.*, 0·5 to 1 ml of approx. *M* acetic acid, H_3PO_4, citric acid, $ZnCl_2$, $FeCl_3$, etc., does not affect the results. The theory of the method is discussed. (*Cf. Anal. Abstr.*, 1955, **2**, 3307, 3308.)

W. J. BAKER

1640. **Flame-spectrochemical analysis. II. Micro-determination of calcium and magnesium.** Shigerô Ikeda (Inst. of Steel, Tohoku Univ., Katabiracho, Sendai, Japan). *J. Chem. Soc. Japan, Pure Chem. Sect.*, 1955, **76** (7), 783–787.—The intensity of flame spectra of Ca (0·1 to 1·0 μg per ml) (551 mμ) and Mg (1·0 to 10 μg) (371 mμ) increases with the addition of lower alcohols, including methanol, ethanol and *iso*propyl alcohol, and with increase of the concn. of the alcohol. The concn. of acid should be kept constant (HCl, <3 *N*; HNO_3, $<0{\cdot}5$ *N*; H_2SO_4, $<0{\cdot}2$ *N*). The influence of other ingredients in the sample soln. is almost the same as in the absence of alcohol. The sensitivity of the quant. analysis of Ca and Mg can be doubled by the use of 50 per cent. methanol, with an error of ± 3 per cent.

K. SAITO

1641. **Rhodizonate as reagent for barium, strontium and calcium.** A. J. Llacer (Dirección Nacional de Química, Rosario, Argentina). *Mikrochim. Acta*, 1955, (5–6), 921–926.—The specificity and sensitivity of the spot tests for barium and strontium are improved by treating the brownish-red rhodizonate spot with a solution of dimethylamine hydrochloride. A change in colour to bright red indicates the presence of barium, and a change to violet - blue indicates strontium. Calcium can be identified by the separation of characteristic crystals after adding a solution of rhodizonic and benzoic acids to a solution of Ca^{++}. A scheme for the separation and estimation of Ba, Sr, Ca and Mg is described.

C. A. SLATER

1642. **Spectrophotometric titration of milligram quantities of barium.** K. Rowley, R. W. Stoenner and L. Gordon (Brookhaven Nat. Lab., Upton, Long Island, N.Y., U.S.A.). *Anal. Chem.*, 1956, **28** (1), 136.—Barium (0·1 to 5 mg) in neutral or faintly acid soln. is treated with methanol, a buffer soln. and a little Eriochrome black T. EDTA (disodium salt) standard soln. is run in and the titration is followed spectrophotometrically at 650 mμ. The concn. of Ba^{++} is derived from standards after a blank correction.

D. A. PANTONY

1643. **Chemical analysis by the use of EDTA complexes. I. Separation of barium and lead with ion-exchange resin.** Tomitsugu Taketatsu (Kyushu Univ., Fukuoka, Japan). *J. Chem. Soc. Japan, Pure Chem. Sect.*, 1955, **76** (7), 756–757.—When a mixed soln. of EDTA complexes of Ba and Pb is passed through a column of Amberlite IR-120 (1 to 1·5 ml per min.), Ba is quantitatively adsorbed on the column from an aq. soln. of pH $<5{\cdot}3$, whilst Pb is not adsorbed from a soln. of pH $>3{\cdot}3$. A satisfactory separation of Ba and Pb is effected by passing a sample soln. containing 0·3 g of EDTA (disodium salt) (pH 4·0 to 4·5) through the column. Barium is eluted from the column with a soln. containing ammonium acetate (2·5 g per 100 ml) and aq. NH_3 (5 ml of conc. aq. NH_3 soln. per 100 ml) (pH 10·8). Both Pb and Ba are titrated in the eluates with $MgCl_2$ soln., with Eriochrome black T as indicator.

K. SAITO

1644. **Determination of zinc by the method of internal electrolysis.** E. F. Speranskaya and M. T. Kozlovskiĭ. *Uchenye Zapiski Kazakh. Gosudarst. Univ.*, 1954, **16**, 86–93; *Referativnyĭ Zh., Khim.*, 1955, Abstr. No. 18,983.—With an anode of sodium amalgam, Zn is separated quant. from alkaline solutions by means of internal electrolysis. The quality of the deposit is improved by the introduction of lead salts. The effect of certain salts is studied. Aluminium is unsuitable as material for the anode.

G. S. SMITH

1645. **New procedure for the detection and determination of zinc by means of fluorescence.** Z. Holzbecher (Ústav anal. chem., Vysoká škola chem.-tech., Prague, Czechoslovakia). *Chem. Listy*, 1955, **49** (5), 684–692.—The fluorescence properties under u.v. light of the zinc salts of a number of salicylaldehyde condensation products have been studied. Of these, the semicarbazone, and especially the acetylhydrazone, are excellent reagents for the detection and determination of Zn. The determination is possible in the concn. range of 5 to 500 μg per 50 ml of soln. at pH 3, or of 5 to 50 μg at pH 6. Aluminium and Fe must be screened by means of 3 per cent. aq. NaF; Li, Na, K, NH_4^+, Sr and Ba in amounts >500 μg per 50 ml do not interfere. *Determination of Zn in Zn acetate*—To the sample (5 ml) containing up to 1·31 mg of Zn add 0·1 *M* acetate buffer (10 ml), followed by ethanol (10 ml) and a 0·01 *M* ethanolic soln. of the reagent (0·5 ml). Dilute to 50 ml with ethanol and measure the fluorescence.

G. GLASER

1646. **Polarographic determination of zinc in copper sulphate.** J. W. Menary (Res. Dept., African Explosives and Chem. Industries Ltd., P.O. Northrand, Transvaal). *Analyst*, 1955, **80**, 908–909.—In the determination of impurities in technical $CuSO_4$ a rapid polarographic method proved satisfactory for the determination of Zn. *Procedure*—The $CuSO_4.5H_2O$ (0·5 g) is boiled with 10 ml of 3·5 *M* NaOH for 5 min. To the cooled liquid are added 15 ml of *M* NH_4Cl - 5 *M* aq. NH_3 soln. and 2 ml of freshly prepared 0·5 per cent. gelatin soln. and the vol. is adjusted to 50 ml with water. The polarogram is recorded in the usual way. The Zn wave occurs at $\simeq -1{\cdot}3$ V against the mercury-pool electrode. Chemically standardised ZnO may be used in the standardisation. By this procedure co-pptn. of Zn with Cu is negligible and 0·1 per cent. of $ZnSO_4$ in $CuSO_4.5H_2O$ can be determined accurately.

A. O. JONES

1647. **New methods for the quantitative determination of cadmium. III. Potentiometric titration with lithium ferrocyanide solution.** A. Basiński and M. Lango (Kopernik Univ., Torun, Poland). *Przem. Chem.*, 1955, **11**, 145–147.—Cadmium sulphate solution (0·1 to 0·5 *N*) is titrated potentiometrically with standard lithium ferrocyanide solution, with platinum and calomel electrodes and magnetic stirring. It is carried out at room temp. in neutral or weakly acidic solution in the absence of K and metals such as Zn, Pb and Cu, which form slightly soluble salts with ferrocyanide. The method is rapid and reliable, the error being less than 0·2 per cent.

A. O. JAKUBOVIC

1648. **Extraction of mercury (I) and (II) and subsequent spot-test detection with dithizone.** P. W. West and McG. A. Duff (Louisiana State Univ., Baton Rouge, U.S.A.). *Mikrochim. Acta*, 1955, (5–6), 987–991.—One drop of the test solution

is mixed thoroughly with 10 drops of trichloroacetic acid (3 *M*), 3 drops of oxalic acid (10 per cent.) and *n*-butyl acetate (1 ml). The separated non-aqueous phase is placed on a white spotting tile and 0·002 per cent. dithizone in chloroform (2 to 3 drops) is added. An orange colour indicates mercury; 1 μg in 1 ml is detectable. Gold (tervalent), halides, cyanide, thiocyanate and sulphide interfere.
C. A. SLATER

1649. **Ascorbimetric determination of mercuric ions.** L. Erdey and I. Buzás (Tech. Univ., Budapest). *Acta Chim. Acad. Sci. Hung.*, 1955, **8** (1–3), 263–270.—Ascorbic acid (**I**) reduces Hg^{++} soln. to Hg metal, and Hg^{++} can thus be determined titrimetrically at pH 2 to 3 with 4-amino-4′-methoxydiphenylamine (**II**) as redox indicator. *Procedure*—Neutralise the Cl'-free soln. containing Hg^{++} (50 to 500 mg) with NaOH, add 0·1 *N* HNO_3 (30 ml) and dilute with H_2O to $\simeq$100 ml. Warm to 60° C and add 1 per cent. **II** (0·1 ml) and titrate with 0·1 *N* **I**; the indicator is temporarily decolorised at the halfway (Hg^{+}) stage and thus permits prediction of the approx. end-point. In the presence of Cl' the reaction proceeds to HgCl only and the method is modified as follows. Treat the Hg^{++} soln. with KNO_3 (2 to 3 g) and **I** (0·5 to 1 g), warm the suspension for 10 to 15 min. and collect the Hg metal. Re-dissolve the Hg in hot HNO_3 (1 + 1) (10 ml), add 0·1 *N* $KMnO_4$ (to remove NO_2'), then 3 per cent. H_2O_2 (1 to 2 ml, to remove MnO_4') and finally warm to remove H_2O_2. Then neutralise the soln. and continue as above. The method permits 10 mg of Hg to be determined with high accuracy (especially when acetate buffer is added near the end-point) and the reaction proceeds satisfactorily in 0·01 *N* soln.
J. P. STERN

1650. **A study of three new colorimetric reagents for trace quantities of boron.** R. L. Grob (Univ. Virginia, U.S.A.). *Dissert. Abstr.*, 1955, **15** (10), 1716.—The three reagents are (5-benzamido-1-anthraquinolyl) - (6 - chloro - 1 - anthraquinolyl) - amine (**I**), 1 - anthraquinolyl - (5 - *p* - toluidino - 1 - anthraquinolyl)amine (**II**) and tetrabromochrysazin (**III**). A complex between boron and the reagent is formed in 96 per cent. H_2SO_4. Variables affecting the complex formation are time of heating, temp., concn. of H_2SO_4, and the presence of diverse ions. Solutions of all three compounds follow Beer's law over the range of boron indicated: **I**, 0 to 0·5 μg at 635 mμ; **II**, 0 to 0·4 μg at 720 mμ; and **III**, 0 to 1 μg at 540 mμ. The sensitivities of **I**, **II** and **III** are 1 in 3×10^8, 1 in 2×10^8 and 1 in 5×10^7, respectively.
O. M. WHITTON

1651. **Determination of the small boron content of gas black.** Z. Nagy (Debrecen Phys. Res. Inst. Hungarian Acad. Sci.). *Magyar Kém. Foly.*, 1955, **61** (11), 351–353.—The sample was compressed to about one-fifth of its original vol. and was then burnt at 700° C in an electric oven. (The ash content was $\simeq$ 0·1 to 0·5 per cent.) After qual. spectrographic analysis, the boron content was determined by continuous addition. To samples of identical wt., containing the unknown, the constituent to be determined was added in known and increasing amounts. From the series of lines thus obtained the concn. of the unknown could be determined, by extrapolation or approximation. The auxiliary electrode was electrolytic copper; 10-mg samples of the unknown were used and 0 to 9 μg of boron was added. Excitation was by an a.c. arc. A Zeiss Q24 spectrograph was used, with a 15 to 20-μ slit. The lines studied were B 2496·8 and Cu 2492·12 A. For a low boron content, with the Seidl transformation, 10^{-4} g per cent. can be determined, *i.e.*, 10^{-6} g per cent., on the original gas black. The sensitivity is about half of this value.
A. G. PETO

1652. **Colorimetric determination of decaborane using NN-diethylnicotinamide.** D. L. Hill, E. I. Gipson and J. F. Heacock (Army Chem. Center, Md., U.S.A.). *Anal. Chem.*, 1956, **28** (1), 133–134.—Decaborane (20 to 240 μg per ml) in water or *cyclo*hexane is treated with an excess of NN-diethylnicotinamide (nikethamide) in water. After 90 min. the orange - red colour intensity is measured at 435 mμ and the decaborane concn. is derived from standards. Boric acid, boron salts, proteins, diborane and pentaborane do not interfere.
D. A. PANTONY

1653. **General method for the determination of aluminium with alizarin as indicator.** A. K. Babko and T. N. Nazarchuk. *Ukr. Khim. Zh.*, 1954, **20** (6), 678–683.—To the test solution are added 0·5 ml of a saturated ethanolic solution of alizarin and 25 ml of aq. 0·6 *N* NaF. If the solution is acid, it is neutralised with 0·5 *N* NaOH to give a blue - violet colour. Then 0·1 *M* acetic acid is added dropwise until the colour is yellow, followed by 25 ml of a buffer solution [a mixture (3:2) of 0·1 *M* acetic acid and aq. 0·1 *M* NH_3]. The solution is heated to between 80° and 90° C and saturated with NaCl, then titrated while hot with standard $KAl(SO_4)_2$ soln. until the colour changes from yellow to pink. The NaF solution is standardised in a similar manner. Tests on solutions containing 10 to 60 mg of Al showed a probable error of $\pm$ 1 per cent. Iron interferes but may be separated by pptn. with $K_4Fe(CN)_6$, followed by addition of a slight excess of $ZnCl_2$ or $MnSO_4$ soln. (This means of separation is not reliable if Fe is in very large excess.) The method was successfully applied to the determination of Al in bauxite. The sample was fused with Na_2CO_3 and borax, dissolved in H_2SO_4, the SiO_2 filtered off, the solution nearly neutralised with NaOH, the Fe separated and the Al titrated as described above. Calcium and Mg need not be separated if present in small amount.
F. W. KIRKBRIDE

1654. **Fluorimetric micro-determination of gallium with Solochrome dyes.** I.-M. Ladenbauer, J. Korkisch and F. Hecht (Chem. Univ. Lab., Vienna). *Mikrochim. Acta*, 1955, (5–6), 1076–1085.—Two methods are described which are based on the increase in fluorescence, proportional to the gallium concentration, in the presence of Solochrome red ERS and Solochrome black WEFA. The determination of 0·1 to 8 μg of gallium in 10 ml of solution is possible, with an accuracy of $\pm$ 5 per cent.
C. A. SLATER

1655. **Titration of gallium with ferrocyanide. Application of dead-stop end-point.** N. R. Fetter and D. F. Swinehart (Univ. Oregon, Eugene, U.S.A.). *Anal. Chem.*, 1956, **28** (1), 122–123.—The formation of $Ga_4[Fe(CN)_6]_3$ is the basis of a titrimetric determination of $Ga^{\cdots}$ with $Fe(CN)_6^{''''}$. The titration is conducted at 50° C and at pH 2·0 against an e.m.f. of 230 mV, the dead-stop end-point detection method being used. Titrations at other pH values are examined; Cl', SO_4'', ClO_4' and $NH_4^{\cdot}$ do not interfere.
D. A. PANTONY

1656. **Radiometric determination of a micro amount of thallium with thallium-204 as indicator.** Tomitaro Ishimori and Yoshimasa Takashima (Kyushu Univ., Fukuoka, Japan). *J. Chem. Soc. Japan, Pure Chem. Sect.*, 1955, **76** (8), 858–862.—The extraction of a micro amount of Tl (<10 μg) from natural substances and its determination with hexa-ammino cobalt as precipitant were examined by means of radioactive ^{60}Co and ^{204}Tl. Since the energies of β-rays are different (^{60}Co, 0·306 MeV; ^{204}Tl, 0·765 MeV), the amounts of both elements can be determined by measuring the activity of the ppt. by the use of aluminium foils of various thicknesses. About 46 to 67 per cent. of the total Tl in silicate rocks is recovered as $[Co(NH_3)_6]TlCl_6$; the loss appears to be due to the insufficient extraction of Tl from the layer of CCl_4 with dil. HNO_3. When these two isotopes are used as indicator, Tl (0·3 to 1 μg per g) can be determined with an error <10 per cent. The sample (silicate) is decomposed with H_2SO_4 and HF, containing a small known amount of ^{204}Tl, and filtered. The filtrate is reduced with hydroxylamine hydrochloride, adjusted to pH 10 and extracted with dithizone - CCl_4 soln. The CCl_4 layer is extracted with dil. HNO_3 (1 + 100), oxidised with $KClO_3$ and the Tl is pptd. with $[Co(NH_3)_6]Cl_3$ containing a known amount of ^{60}Co. The ppt. is dissolved in dil. acetic acid and H_2O_2, evaporated to dryness and the radioactivity is measured with an electroscope. K. Saito

1657. **Colorimetric estimation of cerium with sodium catechol - 3:5 - disulphonate [Tiron].** B. Sarma (Nat. Chem. Lab. of India, Poona). *J. Sci. Ind. Res., B, India*, 1955, **14** (10), 538–539.—Some brief preliminary data are given on the colorimetric estimation of cerium with Tiron. This reagent reacts with Ce^{IV} in a nearly neutral or alkaline medium to form a stable and soluble violet complex. One part of Ce can easily be detected in 5,000,000 parts of solution. There is no interference from large amounts of Th, the rare earths, or common anions such as Cl′, NO_3', SO_4'', PO_4''', citrate, tartrate, fluoride and oxalate. Cerium from 0·4 to 100 p.p.m. can be easily estimated with an error of < ±2 per cent. G. C. Jones

1658. **The thermolysis of the neocupferron chelates of yttrium and the rare-earth elements.** W. W. Wendlandt and J. M. Bryant (Texas Tech. College, Lubbock, Texas, U.S.A.). *Anal. Chim. Acta*, 1955, **13** (6), 550–553.—Thermolysis curves have been determined for the neocupferron chelates of the following elements (temp. of final decomp. to oxides are indicated in brackets)—La (605° C), Ce^{III} (460° C), Ce^{IV} (488° C), Pr (648° C), Nd (750° C), Sm (660° C), Gd (490° C) and Y (523° C). W. C. Johnson

1659. **The rapid separation of rare-earth fission products by cation exchange, using lactic acid eluent.** J. G. Cuninghame, M. L. Sizeland, H. H. Willis, J. Eakins and E. R. Mercer (Atomic Energy Res. Estab., Harwell, England). *J. Inorg. and Nuclear Chem.*, 1955, **1**, 163–164.—A small volume of solution, containing 1 mg each of the chlorides of Y, Eu, Sm, Nd, Pr and Ce, is absorbed on a column of Zeo-Karb 225 (20 to 30 cm long, 3 to 4 mm internal diam., particle size 16 to 27 μ), maintained at 87° C. Individual rare-earths are eluted in the order given above with preheated *M* lactate buffer at pH 3·25 ± 0·03, under a pressure of 5 to 15 lb per sq. in., at 10 to 12 ml per hr. The complete separation takes 8 to 9 hr. The limits of individual fractions are recognised by testing with oxalic acid and also by radioactive tracers, *i.e.*, the rare-earth fission products. E. N. Jenkins

1660. **Catalytic detection of cyanide through the benzoin condensation.** F. Feigl and A. Caldas (Lab. Produção Mineral, Min. Agric., Rio de Janeiro). *Mikrochim. Acta*, 1955, (5–6), 992–995.—The test soln. (1 drop) is treated with alkaline benzaldehyde (0·5 ml of benzaldehyde and 0·5 ml of 6 *N* NaOH made up to 5 ml with alcohol) (1 to 2 drops) and the mixture is heated for 2 to 5 min. on a steam-bath. *o*-Dinitrobenzene in benzene (5 per cent.) (1 drop) is added and the heating is continued. In the presence of cyanide an intense violet colour develops in 0·5 to 3 min. About 0·01 μg of cyanide can be detected at a concn. of 1 in 5×10^6. C. A. Slater

1661. **Some works laboratory experiences with the analysis of synthetic silicates.** E. Hoekstra and F. C. van Dorp (W. Smit & Co. Ltd., Nijmegen, Netherlands). *Chem. Weekbl.*, 1955, **51** (50), 895–900.—The adaptation of known gravimetric or volumetric methods to the analysis of synthetic silicates which are employed in certain welding processes is described. These silicates vary from related products, such as glass and enamel, by higher solubility in strong acids and high content of Mn. The determination of Si, Al, Fe, Ti, Mn, Ca, Mg, K, Na, F, P and S is given. H. A. Fisher

1662. **Spectrophotometry. XII. An improvement for the colorimetric determination of germanium with phenylfluorone.** Yoshinaga Oka and Takuji Kanno (Inst. of Mineral Dressing and Metallurgy, Tohoku Univ., Nagamachi, Sendai). *J. Chem. Soc. Japan, Pure Chem. Sect.*, 1955, **76** (8), 874–877.—The reproducibility of the colorimetric determination of Ge with phenylfluorone (**I**) (Cluley, *Analyst*, 1951, **76**, 523) was examined and it was found that the variation is mainly due to the difference in degree of coagulation of the Ge - **I** complex. The attempt to extract the coloured complex with an organic solvent met with little success. When poly(vinyl alcohol) (0·25 per cent. aq. soln., 5 ml for 50 ml) is used as a protective colloid instead of gum acacia, the extinction at 505 to 520 mμ remains constant for >3 hr. and is proportional to the concn. of Ge for up to 60 μg per 50 ml. The improved method decreased the standard deviation to ±0·005 per cent. K. Saito

1663. **Spectrographic determination of germanium in coal.** G. Szádeczky-Kardoss and I. Benkő (Univ. Chem. Industry, Veszprém, Hungary). *Acta Chim. Acad. Sci. Hung.*, 1955, **8** (1–3), 241–262.—This is an English version of the paper published in *Magyar Kém. Foly.*, 1955, **61**, 225. (See *Anal. Abstr.*, 1956, **3**, 366.) J. P. Stern

1664. **Colorimetric estimation of tin using molybdophosphoric acid reagent.** N. Subramanian and P. B. Janardhanan (A.C. Coll. Technol., Univ. of Madras, India). *J. Sci. Ind. Res., B, India*, 1955, **14** (10), 523–526.—The colorimetric estimation of Sn with molybdophosphoric acid has been critically examined. The colour development with this reagent is instantaneous and permanent up to 24 hr. A method is described in which the usual extraction of molybdenum blue with solvent, before the measurement of colour, can be eliminated.

provided that other colour-producing and interfering elements such as Cu, Ti, Fe and C are absent. *Procedure*—A weighed quantity of the alloy is transferred to a conical flask, and covered with 50 ml of 66 per cent. v/v HCl and 5 ml of a 5 per cent. $SbCl_3$ solution. A pinch of Na_2CO_3 is added to produce an atmosphere of CO_2. The flask is fitted with a tube bent twice at right angles, the free end of which dips into a solution of Na_2CO_3 to prevent entry of air into the system. The assembly is heated on a boiling-water bath until dissolution of the alloy is complete. The cooled solution is made up to 100 ml. The following precautions are necessary: addition of a piece of Zn during dissolution of the alloy, the use of HCl free from Cl, and of boiled and cooled water to make up the solutions. One to 10 ml of solution prepared as above is transferred to a 50-ml flask and the volume made up with the reagent. The intensity of colour is measured within 5 min. after addition of the reagent, in a photoelectric colorimeter, with a 10-mm cuvette. The limiting concn. of Sn which can be estimated by this method are 2 and 35 p.p.m. G. C. JONES

1665. **Semi-quantitative determination of micro amounts of tin in [water from] hot springs.** Nagao Ikeda (Tokyo Univ., Hongo, Japan). *J. Chem. Soc. Japan, Pure Chem. Sect.*, 1955, **76** (9), 1011–1013.—A small amount of Sn (0·5 to 100 μg) in water from hot springs (sample, 1 to 20 litres) is extracted with dithizone - CCl_4 soln. at pH 5 to 6 in the presence of tartrate. The organic layer is evaporated to dryness in a silica dish and decomposed with a small amount of HNO_3. Sodium nitrate (10 mg) is added to the product and the mixture is heated. The residue is placed on a carbon electrode and a d.c. arc is struck. The intensities of the spectral lines of Sn are compared with those of the standard samples and the amount is roughly estimated. K. SAITO

1666. **Determination of tin in siderurgical products.** J. Migeon. *Chim. Anal.*, 1955, **37** (12), 416–421.—The sample (2 g for less than 0·02 per cent. of Sn; 1 g for more) is treated with conc. HCl (30 ml) and oxidised with H_2O_2 (a few drops). After the addition of $HClO_4$ (15 ml), the soln. is distilled (apparatus described) in a stream of CO_2 and the distillate is collected in water (50 ml). When the perchloric vapours appear, heating is stopped and the solution is allowed to cool to below 100° C (in an atmosphere of CO_2). Concentrated HCl (10 ml) is added and distillation is continued. The whole process is repeated four times. To the distillate is added aq. NaOH (40 per cent.) (10 ml) and the solution is concentrated to <5 ml. Perchloric acid (50 ml) is added and the solution is made up to 100 ml. A 10-ml portion is subjected to polarography and the Sn is calculated by reference to solutions containing known quantities. C. A. SLATER

1667. **Study of interferences in the iodimetry of tin. Its application to ore analysis.** R. A. G. de Carvalho (Lab. Kudi, Porto, Portugal). *Anal. Chim. Acta*, 1956, **14** (1), 28–38.—In the determination of tin in 4 *M* HCl soln. by reduction with aluminium wire followed by titration with 0·15 *N* KI - KIO_3 soln., slightly high results are obtained in the presence of ≃0·02 g of Mo, and low (often variable) results when from 0·05 to 0·1 g of Mo is present, although in this instance the effect is less if the titration is made rapidly. These anomalies are discussed and are ascribed mainly to the presence of Mo^{III} (by reduction of Mo^{V} with $Sn^{\cdot\cdot}$) in soln. (*cf. Anal. Abstr.*, 1954, **1**, 2975). Because $Fe^{\cdot\cdot}$ catalyse the conversion of Mo^{III} into Mo^{V}, they do not lead to low results when present. A modified procedure for the iodimetric determination of Sn in cassiterite and tungsten ores is described. It is based on dissolution of the sample (0·5 g) by conc. HNO_3 (plus conc. HCl if tungsten is present) followed by retention of any sol. Sn in the $Fe(OH)_3$ pptd. on addition of excess of strong aq. NH_3 (an addition of Fe is made if necessary). Tungsten, Mo and Cu remain in the filtrate; the ppt. of non-attacked cassiterite plus retained Sn is ignited and fused with Na_2O_2 to obtain a soln. for reduction and titration. W. J. BAKER

1668. **Thermometric titration of stannic chloride.** S. T. Zenchelsky, J. Periale and J. C. Cobb (Rutgers Univ., New Brunswick, N.J., U.S.A.). *Anal. Chem.*, 1956, **28** (1), 67–69.—Stannic chloride is titrated calorimetrically with dioxan in benzene, carbon tetrachloride, nitrobenzene or chloroform solution. Allowance having been made for heats of solvation, the stannic chloride is determined to an accuracy within 1 per cent. D. A. PANTONY

1669. **Micro-gravimetric determination of lead with thionalide.** C. Cimerman and M. Ariel (Inst. Technol., Haifa, Israel). *Anal. Chim. Acta*, 1956, **14** (1), 48–52.—The micro-method described depends on pptn. of $Pb^{\cdot\cdot}$ from an alkaline soln. containing KCN and Na K tartrate by a soln. of thionalide (thioglycollic acid 2-naphthylamide) in acetone. *Procedure*—To 3 ml of the slightly acid soln. of the sample (≃ 2 to 5 mg of Pb) add ≃ 50 mg of Na K tartrate, stir well, add one drop of phenolphthalein (1 per cent. ethanol soln.) and then 2 *N* NaOH soln. dropwise until the pptd. $Pb(OH)_2$ just redissolves giving a red soln. Add ≃100 mg of KCN, heat the solution to 80° C and then add freshly prepared thionalide soln. (30 to 40 mg in 1 to 2 ml of acetone) dropwise from a pipette, with constant stirring. Heat the mixture carefully for 1 to 2 min. at 80° C, allow the ppt. to settle (1 to 2 min.) and filter (low suction) through a porcelain filter-stick (55 mm high, stem-diameter 3 to 4 mm., overall diameter 11 mm). Wash the beaker, rod, filter-stick and ppt. with acetone - H_2O (1 + 1), H_2O, and then acetone - H_2O, using ≃ 15 ml in all, dry at 110° C and weigh. Wt. of ppt. × 0·3239 = Pb. The accuracy is within ± 0·6 per cent. and one determination takes ≃2½ hr. W. J. BAKER

1670. **Gravimetric determination of lead as picrolonate in the presence of zinc, and the influence of other metal ions.** Hiromu Imai (Nichia Chem. Ind. Ltd., Higashi-Yodogawa, Osaka, Japan). *J. Chem. Soc. Japan, Pure Chem. Sect.*, 1955, **76** (7), 770–774.—The gravimetric determination of Pb as picrolonate by Hecht *et al.* (*Z. anorg. Chem.*, 1933, **95**, 152) was examined in the presence of an excess of Zn. Ammonium nitrate is a good masking agent for <22 mg of Zn, when the Pb picrolonate is produced with a large excess of picrolonic acid (1 g per litre, 20 ml for <5 mg of Pb) at pH 2 to 6. No interference results from the presence of Cd (<130 mg), Al (<60 mg), Mn (<40 mg), Ag (<20 mg), Cr (<15 mg) and Hg (<10 mg). K. SAITO

1671. **Determination of hydrogen in titanium and titanium alloys.** M. Codell and G. Norwitz (Frankford Arsenal, Philadelphia, Pa., U.S.A.). *Anal. Chem.*, 1956, **28** (1), 106–109.—The titanium metal

or alloy is ignited under O in the presence of a lead flux. The resulting H_2O is collected in a weighed tube of $MgClO_4$ and the hydrogen content is determined after allowance has been made for a blank.

D. A. PANTONY

1672. **The determination of hydrogen chloride in titanium tetrachloride.** T. R. Ingraham (Div. of Mineral Dressing and Process Metallurgy, Mines Branch, Ottawa, Canada). *Canad. J. Chem.*, 1955, **33** (11), 1731–1733.—Titanium tetrachloride (25 ml) is added against a countercurrent of He to a tube connected to a mercury pressure gauge and a 250-ml bulb. The $TiCl_4$ is frozen by liquid air and the apparatus is evacuated. The $TiCl_4$ is allowed to melt, and then is refrozen in a slurry of solid CO_2 in a CCl_4 - $CHCl_3$ mixture. The pressure of the HCl evolved during the melting is measured. Silicone stopcock grease is used. The Hg does not require protection. Repeat analyses on a single batch of $TiCl_4$ gave 0·032, 0·034 and 0·030 per cent. of HCl. A sample saturated with HCl at room temp. contained 0·117 per cent. of HCl.

A. B. DENSHAM

1673. **An improved volumetric method for zirconium.** G. W. C. Milner and J. W. Edwards (Atomic Energy Res. Estab., Harwell, Berks., England). *Analyst*, 1955, **80**, 879–884.—In the method of Milner *et al.* (*Anal. Abstr.*, 1954, **1**, 2947) for the determination of Zr by addition to the test soln. of a slight excess of 0·02 *M* EDTA and back-titration of the excess with standard $Fe^{\cdots}$ soln., with salicylic acid as indicator, the accuracy is no better than ±1 per cent., the error being mainly personal error in observing the end-point. In the improved procedure now described the end-point is determined spectrophotometrically. The E.E.L. absorptiometer is suitable for this purpose since the cell carriage can be replaced by a titration vessel with stirrer. With the test soln. and an Ilford No. 605 filter the extinction is adjusted to zero and after each addition of 0·02 *M* $Fe^{\cdots}$ soln. the extinction is recorded. In the graph of the extinction against the vol. of titrant the end-point is shown as the intersection of two straight lines. Sodium salicylate is used as indicator in the pH range 3 to 7 and potassium benzohydroxamate in the range 1·8 to 3·3. Up to 100 mg of Zr can be determined with an accuracy within ±0·6 per cent. Uranyl ions and $Ce^{\cdots}$ do not interfere and the method is thus applicable to uranium - zirconium and cerium-based alloys.

A. O. JONES

1674. **Electrometric titration of zirconium with the aid of an indicator.** Yu. I. Usatenko and G. E. Bekleshova. *Ukr. Khim. Zh.*, 1954, **20** (6), 690–692.—To carry out a determination, 15 ml of the test solution, acidified with HCl and containing $>0·025$ g of ZrO_2, are placed in a 100-ml beaker, and 3 g of NaCl and 20 ml of ethanol are added. The solution is adjusted to a pH of $\simeq 2·2$ by adding aq. NH_3 (faint pink tinge of methyl orange), a platinum electrode and an agar bridge leading to a S.C.E. are inserted, and 0·1 ml of *M* $FeCl_3$ is added. With the platinum electrode rotating at $\simeq 900$ r.p.m., the solution is titrated at room temp. with 4 per cent. NaF from a micro-burette until a galvanometer (resistance 760 ohms) in series with the electrodes indicates that the zero-point has been passed. If the acidity decreases during the titration, dilute HCl is added dropwise to restore the pink colour. The titration curve is plotted so that the quantity of NaF required can be read off to 0·001 ml. The NaF solution is standardised gravimetrically by pptn. of PbClF. The titration is not interfered with by Ca, Mg, Ti, Cr or Fe^{II} nor by silicic acid, but Al and Be are included in the titre. A correction of 0·065 ml is made for the NaF consumed by the iron indicator.

F. W. KIRKBRIDE

1675. **The direct weighing of zirconium tetramandelate.** R. B. Hahn and E. S. Baginski (Wayne Univ., Detroit, Mich., U.S.A.). *Anal. Chim. Acta*, 1956, **14** (1), 45–47.—In the determination of Zr by pptn. with mandelic acid (*cf. Anal. Abstr.*, 1954, **1**, 909) errors are introduced by the presence of basic salts in the Zr tetramandelate ppt., which is usually weighed directly. Reproducible and more accurate weighings are obtained when the pptn. is made at 85° to 90° C from <5 *M* HCl by dropwise addition of mandelic acid, filtering the soln. when cool and washing the ppt. with water saturated with Zr tetramandelate, followed by 95 per cent. ethanol and finally ether. No correction factor is needed.

W. J. BAKER

1676. **Emission spectrometric determination of low percentages of zirconium in hafnium.** L. S. Gray, jun., and V. A. Fassel (Iowa State College, Ames, Iowa, U.S.A.). *Anal. Chem.*, 1956, **28** (1), 18–21.—A 12-amp. d.c. arc technique is used to determine Zr in the range 0·001 to 0·2 per cent. in Hf with a precision corresponding to a coeff. of variation of 8 to 9 per cent. of content. Greater precision is attained by using an overdamped 25-μF discharge from a Multisource on a briquetted sample, when the coeff. of variation is of the order of 3 to 5 per cent. of content over the range 0·01 to 0·5 per cent. of Zr.

P. T. BEALE

1677. **Photometric micro-determination of zirconium in iron and steel.** Kiichi Narita (Kobe Steel Manufacturing Co., Ltd., Nada-ku, Kobe, Japan). *J. Chem. Soc. Japan, Pure Chem. Sect.*, 1955, **76** (9), 1026–1030.—The application of the colorimetric determination of Zr with alizarin red S (**I**) to the determination of Zr (0·001 to 0·03 per cent.) in iron and steel was studied, with special reference to the elimination of effects due to ferric iron. The extinction of the Zr - **I** complex is markedly decreased by the presence of an excess of SO_4'' ($>0·05$ g per 100 ml). The presence of Fe in amounts <25 mg does not interfere with the colorimetry if thioglycollic acid is used for masking. *Procedure*—The sample (1 g) is dissolved in 6 *N* HCl (20 ml) and oxidised with HNO_3, the excess of which is removed by heating with HCl. The product is dissolved in 6 *N* HCl (20 ml), filtered and the filtrate is shaken with ether (50 ml). Zirconium is pptd. with aq. NH_3 soln., the ppt. is ignited together with the insol. residue of the sample (SiO_2, C, ZrO_2, etc.), treated with HF and H_2SO_4, and ignited. The product is fused with Na_2CO_3 (2 g) (to remove traces of P), extracted with water and filtered. The residue is ignited, fused with $Na_2S_2O_7$ (1 g), dissolved in 5 per cent. HCl and treated with H_2O_2 (3 per cent.) (10 ml), to remove Ti, and NaOH to produce zirconium hydroxide. The ppt. is dissolved in 6 *N* HCl (10 ml) and aq. NH_3 soln. is added to bring the pH to 1. The soln. is treated with thioglycollic acid (10 per cent.) (1 ml) and **I** (0·05 per cent.) (3 ml) and made up to 100 ml. The extinction at 520 mμ is measured after 1 hr. The results agree well with those obtained by gravimetric analysis.

K. SAITO

1678. **Trichlorophenoxyacetic acid as a reagent for the determination of thorium.** S. K. Datta (Gov. Coll., Darjeeling, India). *Anal. Chim. Acta*, 1956, **14** (1), 39–44.—Thorium can be separated effectively from Ca, Ba, Sr, Zn, Co, Ni, Ti, U and cerite earths by quant. pptn. with a hot 0·5 per cent. soln. of 2:4:5-trichlorophenoxyacetic acid (**I**) and weighing the washed and dried (110° C) salt of Th. Unlike the use of 2:4-dichlorophenoxyacetic acid, pptn. is complete at pH 2 and no double pptn. is needed to remove Co, Ni or Cr. Large amounts of U interfere, but Th can be completely separated by double pptn. with **I** from solutions having thoria to U_3O_8 ratios up to 1:25. For solutions having thoria to rare-earth ratios up to 1:40, one pptn. will separate Th completely. Interference is also caused by Zr and Fe, but Fe can be removed by pptn. of Th + Fe with **I** at pH 2·6 and then treating the washed ppt. with just sufficient aq. 5 per cent. ammonium carbonate to form a sol. Th - ammonium carbonate complex. The small amount of Th retained in the residual ppt. of Fe after filtration is recovered by another treatment with ammonium carbonate. The combined filtrates are then boiled with a few ml of conc. HCl, and Th is re-pptd. with **I** at the required pH. The thorium salt of **I** should always be washed with hot 50 per cent. ethanol. (*Cf. Anal. Abstr.*, 1955, **2**, 1788.)

W. J. Baker

1679. **Analytical aspects of some organic compounds. I. Some phenolic acids in the estimation of thorium and zirconium.** S. K. Datta (Chem. Lab., Gov. Coll., Darjeeling, India). *J. Indian Chem. Soc.*, 1955, **32** (10), 687–693.—Comparative studies on the estimation of Th and Zr by the following phenolic acids were made: salicylic, 5-nitro-, 5-bromo- and 5-amino-salicylic, 2:4-dihydroxybenzoic, 5-bromo-2:4-dihydroxybenzoic and gallic acids. Thorium is quant. pptd. with all acids at pH >4. The pptn. of Zr is anomalous in respect of pH and does not follow any definable rule. Thorium and Zr were separated by the use of 5-nitrosalicylic and 2:4-dihydroxybenzoic acids; Th was quant. pptd. at pH >4 and Zr at pH 2 to 4. Double pptn. is necessary for complete separation. The separation of Th from rare earths, extraction of Th from monazite sand and separation of Th and Zr from other elements were also studied.

S.C.I. Abstr.

1680. **Indirect colorimetric determination of thorium.** Tôru Nozaki (Ehime Univ., Niihama, Japan). *J. Chem. Soc. Japan, Pure Chem. Sect.*, 1955, **76** (9), 996–998.—Thorium (<1 mg) is pptd. from a 0·1 to 0·2 *N* mineral acid soln. with microcosmic salt (10 per cent., 5 ml) as $Th(HPO_4)_2$; the ppt. is washed with NH_4NO_3 soln. (5 per cent.) and dissolved in 3 *N* HCl. The soln. is neutralised with *N* NaOH to *p*-nitrophenol, treated with ammonium molybdate (1·5 per cent. in 4 *N* HCl) (5 ml) and $SnCl_2$ (0·12 per cent. in 0·04 *N* HCl) (2·5 ml) and made up to 50 ml. The soln. is compared with standard soln. and the amount of Th calculated with an error <2 per cent. The presence of Fe^{++}, Fe^{+++}, Al, Ti and Zr vitiates the determination, but Th can be separated from these ions by passing a 2 *N* HCl soln. through a column of Amberlite IR-120; the column is washed with 2 *N* HCl and eluted with 0·5 *M* oxalic acid soln. The recovery of Th is ≃90 per cent. K. Saito

1681. **Determination of thorium and uranium concentration ratios of Indian rocks and minerals.** V. Seetharam Aithal (Indian Inst. Sci., Bangalore). *J. Sci. Ind. Res., B, India*, 1955, **14** (10), 519–523.—The concentration ratios of Th to U in 10 samples of rocks and minerals have been determined by estimating the range of the α-particles emitted by Th and U with a scintillation counter. The method was based on that of Evans and Finney (*Phys. Rev.*, 1935, **48**, 503) and a scintillation counter E.M.I. VX 5045 was employed. The sample was powdered to 100 mesh and spread over a standard specimen holder. The number of α-particles emitted was measured by keeping the specimen at a known distance from the photo-cathode for 1 hr. The experiment was repeated at a different position. An equation was developed from which the concentration ratio of Th to U could be calculated.

G. C. Jones

1682. **The microchemical identification of condensed phosphates.** A. Simon and E. Steger (Tech. Hochschule, Dresden, Germany). *Naturwissenschaften*, 1955, **42** (22), 604.—Characteristic crystalline precipitates are obtained with hexaamminocobalt trichloride (**I**). A few specks of the phosphate are mixed on the microscope slide with a drop of dilute acetic acid and a drop of a saturated solution of **I**. From dilute solutions, PO_4''' yields rhomboids and P_2O_7'''' similar rhomboid plates; P_3O_{10}''''' are recognised by thin needles either branched or in star-shaped clusters. The more soluble P_3O_9''' (trimetaphosphate) forms more slowly a ppt. of rectangular bars and P_4O_{12}'''' (tetrametaphosphate) gives rise to rhomboid plates that are joined in their diagonal axis.

E. Kawerau

1683. **Quantitative paper-chromatographic [determination of] condensed phosphate.** W. Koberlein and H. Mair-Waldburg (Milk Management Res. and Experimental Inst., Kempten, Germany). *Z. LebensmittUntersuch.*, 1955, **102** (4), 231–235.—The method described is suitable for determining condensed phosphate in aq. soln., or in melted cheese. The method of Grunze and Thilo, with *iso*propyl alcohol (70 ml), water (10 ml), trichloroacetic acid (20 per cent.) (20 ml) and aq. NH_3 (25 per cent.) (0·3 ml) gave good separation. A small paper roll is used to obtain a quick-running and intact ring chromatogram. For quant. evaluation, ferric thiocyanate is recommended as a suitable spray reagent; for qual. examination the dried chromatogram is sprayed with ammonium molybdate in HNO_3, dried at 100° C, treated with a soln. containing $KHSO_3$, Na_2SO_3 and metol and dried at room temp. S.C.I. Abstr.

1684. **Use of complexones in chemical analysis. XLVI. Gravimetric and volumetric determination of arsenate.** M. Malínek and B. Řehák (Hutnická lab., Čsl. akad. věd, Prague, Czechoslovakia). *Chem. Listy*, 1955, **49** (5), 765–767.—*Gravimetric procedure*—Treat the sample containing As^V with solid EDTA (disodium salt) (**I**), add a slight excess of aq. NH_3, dilute the soln. to 100 ml, add to it dropwise a 0·1 *M* soln. of $MgCl_2.6H_2O$ to permanent turbidity, add a further 20 ml of the Mg^{++} soln. and 20 ml of conc. aq. NH_3. Collect the ppt. after 12 hr., wash it with *N* aq. NH_3 containing 2 to 3 per cent. of NH_4NO_3, dry it at 100° C, ignite it for 20 min. at 900° C and weigh as $Mg_2As_2O_7$. *Volumetric procedure*—Dissolve the ppt. of $MgNH_4AsO_4.6H_2O$ in 10 ml of conc. HCl, neutralise the soln., make it weakly acidic with 3 *N* HCl, cool and add an excess (20 ml) of 0·1 *M* **I**. Dilute the soln. to 100 ml, adjust the pH by means of the Schwarzenbach

buffer (350 ml of 25 per cent. aq. NH_3 and 54 g of NH_4Cl in 1 litre of H_2O) and back-titrate the excess of **I** with 0·1 *M* Mg^{++} to Eriochrome black T (1 ml of 0·1 *M* **I** ≡ 7·491 mg of As). Alternatively, reduce the soln. of the ppt. with 2 g of hydrazine sulphate and 0·5 g of KBr, and titrate with 0·1 *M* **I** to Eriochrome black T. G. GLASER

1685. **Polarographic analysis for antimony in glass.** J. P. Williams and T. A. Schwenkler. *J. Amer. Ceram. Soc.*, 1955, **38** (10), 367–369.—Glasses do not contain ions that interfere with normal polarographic methods applied directly to the solution of the glass in a mixture of HF and oxalic acid. *Procedure*—The glass (1 to 0·5 g, containing 0·2 to 1·5 per cent. of Sb_2O_3) is treated with 5 to 10 ml of H_2O, 2 g of oxalic acid and 15 ml of 48 per cent. HF and evaporated to dryness on a steam-bath. The process is repeated with 5 ml of HF. The oxalic acid is expelled at 150° ± 5° C, then two similar treatments are given after the addition of 2 g of oxalic acid, and the residue is dissolved in 50 ml of solution normal to both acids and made up to 100 ml after the addition of 10 ml of 0·1 per cent. solution of gelatin. Measured at 25° C after passing N through the solution for 10 min., the results were within 0·02 per cent. of Sb_2O_3 of those obtained by wet chemical analysis with 21 glasses containing 0·2 to 2·0 per cent. of Sb_2O_3. J. A. SUGDEN

1686. **Determination of trace quantities of bismuth in lead and copper.** D. T. Englis and B. B. Burnett (Univ. Illinois, Urbana, U.S.A.). *Anal. Chim. Acta*, 1955, **13** (6), 574–587.—The Bi is first separated by collection, from aq. soln., on silica flour, and the optimum conditions for collection and recovery have been determined. A method is described for the determination of Bi, down to 0·00002 per cent., in lead. *Procedure*—Dissolve the sample (20 g) in 75 ml of dil. HNO_3 (1 + 3), add 30 ml of conc. HCl and cool to between 0° and 5° C. Filter off the $PbCl_2$ in a Büchner funnel and wash with dil. HCl (1 + 1). To the filtrate and washings add 20 ml of ammonium acetate soln. (20 per cent.) and neutralise with NaOH pellets, cool to room temp. and adjust to a pH of 6·0 to 6·25 with aq. NaOH or dil. HCl. Add 2·5 g of silica flour (Celite or Super Cel), or 1 g if the sample contains ⩾ 0·0005 per cent. of Bi, and set aside for 1 hr. with occasional stirring. Filter off the silica in a Gooch crucible containing an asbestos mat and a layer (3 to 5 mm) of silica, wash it 3 times with water, then extract the Bi with 12·5 ml of hot dil. H_2SO_4 (1 + 20) in two portions, 8 to 9 ml in the first, allowing each portion to flow through by gravity before applying suction. Wash finally with water. To the filtrate and washings add 1 ml of hypophosphorous acid (30 per cent.) and 0·375 g of KI and dilute to 25 ml. Filter, if necessary, through a sintered-glass filter and determine the absorption at 460 mμ on a Beckman DU spectrophotometer. Use a 5-mm cell for contents of Bi < 0·0005 per cent. Prepare a calibration graph with the use of a standard soln. of Bi. Antimony interferes to the extent that 1 mg in 25 ml of soln. shows an absorption of 0·074. A method suitable for the determination of 10 μg to 10 mg of Bi in a 2-g sample of copper is also described. *Procedure*—Dissolve the sample in 20 ml of dil. HNO_3 (1 + 1), separate any insol. matter, fuse it with $K_2S_2O_7$, leach with dil. H_2SO_4 and add the filtrate to the main soln. If enough Pb is present to cause pptn. of $PbSO_4$ the ppt. must be removed. To the soln. add 100 ml of water, 5 g of NH_4Cl and aq. NH_3 to produce a pH of 9. Add 2·5 g of silica flour, or 1 g for contents of Bi ⩾ 100 μg, and complete the analysis as for Bi in lead, but wash the silica with dil. aq. NH_3 (1 + 5) to remove Cu^{++}. Zinc (20 per cent.) and Fe (0·5 per cent.) do not interfere. Lead (0·5 per cent.) reduced the Bi recovery to 90 per cent., probably through occlusion on the ppt. of $PbSO_4$. W. C. JOHNSON

1687. **A ferricyanide - cerimetric determination of vanadium.** B. Suseela (Hindu Univ., Benares, India). *Anal. Chim. Acta*, 1955, **13** (6), 543–545.—A solution of $VOSO_4$ is treated with a 1·5 to 2-fold excess of 0·1 *N* $K_3Fe(CN)_6$ and sufficient aq. NaOH to produce a concn. between 0·5 and 1·0 *N*. After 30 min. the soln. is diluted to 250 ml, a 5 to 60-ml aliquot (≡ 0·023 to 0·27 g of V) is treated with dil. H_2SO_4 (1 + 3) until a green ppt. appears, and the $Fe(CN)_6^{''''}$ produced are titrated with 0·04 to 0·1 *N* $Ce(SO_4)_2$, ferroin being used as indicator.
W. C. JOHNSON

1688. **Amperometric determination of vanadium.** C. L. Rulfs, J. J. Lagowski and R. E. Bahor (Univ. Michigan, Ann Arbor, Mich., U.S.A.). *Anal. Chem.*, 1956, **28** (1), 84–86.—Vanadium, especially from titanium-based alloys, is converted into V^{V} by standard methods and titrated with standard Fe^{++}. The titration is followed amperometrically with a rotating platinum electrode. Vanadium may be determined to within 1 per cent. in the presence of Ti^{++++}, Fe^{+++}, Mn^{++}, Mo^{VI} and W^{VI}.
D. A. PANTONY

1689. **Study of paper electrophoresis. The separation of niobium and tantalum.** E. Bruninx, J. Eeckhout and J. Gillis (Lab. de Chim. Anal., Univ. of Ghent, Belgium). *Anal. Chim. Acta*, 1956, **14** (1), 74–88.—Micro amounts (10 to 50 μg) of Nb and Ta can be separated completely as oxalic acid complexes by paper electrophoresis in a citric acid - potassium citrate buffer (pH 3·42, ionic strength 0·4) at a current intensity of 4 mA (220 V) for 4 to 8 hr. The paper strip (Whatman No. 1, 30 cm × 3 cm) is placed horizontally on a glass plate (the usual top plate being omitted) inside a closed container saturated with water-vapour. The paper is conditioned for 40 min. after impregnation with the buffer soln.; a diaphragm prevents diffusion of the products of electrolysis through the paper. The different factors influencing the displacement of ions by paper electrophoresis are discussed theoretically and from the results of electrophoretic measurements. If the saturation currents are minimised and current and ionic strength kept const., the displacement is approx. proportional to the time of electrophoresis, so that more difficult separations can be made by using longer strips.
W. J. BAKER

1690. **The determination of radioactivity due to fission-product niobium.** D. F. C. Morris and D. Scargill (Atomic Energy Res. Estab., Harwell, England). *Anal. Chim. Acta*, 1956, **14** (1), 57–61.—A rapid radiochemical method of determining Nb in $UO_2(NO_3)_2$ solutions prepared from irradiated U "cooled" for various periods is described. The sample (1 to 10 ml) is treated by the Glendenin method (boiling with HNO_3 and oxalic acid in the presence of Nb oxalate as carrier) to remove Nb from other fission products. The centrifuged pptd. niobic acid is then purified by extraction with tri-*n*-butyl phosphate from HF - H_2SO_4 solution, followed by re-pptn. of niobic acid with aq. NH_3. After centrifuging (1 min.), the organic phase is discarded and

the aq. phase (washed with light petroleum) is centrifuged (1 min.) to obtain the ppt. The ppt. is finally ignited at 800° C for 15 min. and then weighed as Nb_2O_5 (yield is $\simeq$60 per cent.). The activity is measured by counting on a γ-scintillation counter through a standard aluminium - lead sandwich. The procedure takes $\simeq 1\frac{1}{2}$ hr.; radiochemical purity and separation from Cs, Ce, Pr, Ru, Rh, Mo, Tc and Zr are adequate. The standard deviation is $\gtrsim \pm 2$ per cent. provided that the "cooling" time of the sample since irradiation is $\lesssim$30 days.

W. J. BAKER

1691. **Analytical application of the reaction between sulphur and an alkali cyanide.** J. K. Bartlett (Stanford Univ., U.S.A.). *Dissert. Abstr.*, 1955, **15** (6), 955.—Sulphur is quant. converted into thiocyanate by alkali cyanide in 95 per cent. acetone and can be determined by adding $FeCl_3$ and measuring the extinction at 465 mμ. Alternatively, S can be determined in acetone soln. by titrating with standard NaCN soln. in 80 per cent. *iso*propyl alcohol; an acid - base indicator or a potentiometric method is used to detect the end-point. Mercaptans and alkali hydroxides interfere. Cyanide may be determined by titration in pyridine - water (1:2) with standard soln. of S in pyridine until a turbidity appears. N. E.

1692. **A reagent for sulphur dioxide.** H. F. Liddell (Min. Supply, Chem. Defence Exptl. Estab., Porton, Wilts., England). *Analyst*, 1955, **80**, 901.—Astrazone pink FG (Bayer) was found to be a satisfactory reagent for the detection and approx. determination of SO_2 in "smog." A soln. of the dye (0·1 g) in 20 ml of hot water is cooled and mixed with 50 ml of a 10 per cent. soln. of $NaHCO_3$ and 25 ml of glycerol and the vol. is adjusted to 100 ml. A drop is applied to filter-paper held between two transparent plastic sheets, each perforated with a circular hole of diam. 0·11 in. The plastic holder is attached to the inlet of a hand pump (described in the Home Office pamphlets on "Methods of Detection of Toxic Gases in Industry"). Each stroke of the pump draws a known vol. of air through the paper and the pump is operated until the dye stain is bleached to a predetermined extent, the vol. of air producing this effect being noted.

A. O. JONES

1693. **Determination of the sulphur content of nickel.** T. R. Andrew and C. H. R. Gentry. *Vide*, 1955, **10**, 394–400.—Dissolve the sample (0·5 to 1·0 g of Ni) in 6 *N* HCl. Lead the liberated H_2S into the absorption vessel by a stream of H or N which has been freed from H_2S by bubbling through $KMnO_4$ and NaOH soln. The absorption vessel contains an ammoniacal soln. of $CdCl_2$; it should be kept in a blackened container to prevent oxidation of CdS to $CdSO_4$. The absorbent contains 0·2 *M* NH_4Cl, 1·4 *M* aq. NH_3, 0·005 per cent. of gelatin and 0·5 mg of Cd as $CdCl_2$ in 10 ml of soln. The residual Cd in soln. is determined polarographically and the S is calculated from the result.

CHEM. ABSTR.

1694. **Volumetric determination of selenium.** S. Barabas and W. C. Cooper (Canadian Copper Refiners, Ltd., Montreal East, Canada). *Anal. Chem.*, 1956, **28** (1), 129–130.—Selenium is converted into SeO_3'' in H_2SO_4 soln., and this, in the presence of HPO_4'', is converted into SeO_4'' with an excess of standard MnO_4'. The excess of this is back-titrated with standard Fe$^{••}$ soln.

D. A. PANTONY

1695. **Research on "flotation" analysis. II. Separation and determination of traces of selenium and tellurium in difficultly soluble substances.** Yukito Yoshida (Tokyo Inst. Tech., Ookayama, Tokyo). *J. Chem. Soc. Japan, Pure Chem. Sect.*, 1955, **76** (8), 856–858.—When a sample containing Te and Se is fused with a mixture of KOH, KNO_3 and K_2CO_3 (6:12:1) containing $Cd(NO_3)_2$, Se is quantitatively extracted with water, whilst Te remains in the residue. The addition of Cd is essential to keep Te in the residue. The filtrate is made acid and treated with $SnCl_2$ to reduce selenate to selenium, which is collected on the boundary of the aqueous and ether layers and compared with a standard. Tellurium in the residue is dissolved in HCl, shaken with ether to remove Fe (and Au and Tl), reduced to the elementary state with hydrazine hydrochloride and similarly determined.

K. SAITO

1696. **Polarographic determination of selenium and tellurium.** Yoshio Morimoto and Fukunosuke Suzuki (Mitsubishi Metal Mining Co. Ltd., Shinkawasakicho, Osaka, Japan). *J. Electrochem. Soc. Japan*, 1955, **23** (10), 544–547.—The polarographic determination of Se and Te by Lingane *et al.* (*J. Amer. Chem. Soc.*, 1947, **70**, 4115; 1949, **71**, 196) was examined with reference to its use for factory control. Optimum concn. of the supporting electrolyte (*M* NH_4Cl, 0·5 *M* aq. NH_3 for 10^{-3} to 10^{-4} *M* Te) and of the max. suppressor are given. The shape of the wave depends on the concn. of NH_3 and gelatin. For a simultaneous determination of Se and Te, the use of a 0·004 to 0·008 per cent. soln. of gelatin appears to be suitable. Lithium citrate (0·5 *M*) makes the measurement of the wave height of Se easier. K. SAITO

1697. **Colour reactions of bivalent chromium.** V. I. Kusnetsov. *Doklady Akad. Nauk SSSR*, 1954, **95** (3), 547–550; *Referativnyĭ Zh., Khim.*, 1955, (9), Abstr. No. 16,566.—The reactions of Cr$^{••}$ with various azo compounds, leading to the formation of intensely coloured complexes of Cr^{III}, are described. The reactions are carried out in soln. weakly acidified with HCl. Soln. of $CrCl_2$ are obtained by reduction of $K_2Cr_2O_7$ soln. in HCl by zinc amalgam. The colours formed are very stable to subsequent acidification with HCl. The mechanism of the reactions is as follows. On adding Cr$^{••}$ soln. to an excess of reagent soln., part of the latter is used for oxidation of Cr$^{••}$ to Cr$^{•••}$ (the oxidising group being $-NO_2$ or $-N:N-$). The Cr$^{•••}$ formed immediately react with a further molecule of reagent, producing a Cr^{III} complex, the soln. of which displays in the acid medium an intense colour because of the strong internal dissociation and, in some cases, additional ionic states. Chromic salts of the corresponding complexes are either not formed, or are formed only after prolonged heating of the Cr$^{•••}$ soln. with the reagent. This is because in soln. of Cr$^{•••}$ salts in water, stable aquo-acid complexes are formed, which react with the organic reagents extremely slowly. Such strong reducing agents as V$^{••}$, Sn$^{••}$, Yb$^{••}$ and $Na_2S_2O_4$ do not give colour reactions with the organic reagents named, because these reducing agents are not in a state to form cyclic salts with strong internal dissociation which are stable in a strongly acid medium.

C. D. KOPKIN

1698. **Titrimetric micro-determination of chromium (VI) and chromium (III) by a catalytic reaction.** G. Almássy and E. Kovács (Med. Univ., Debrecen,

Hungary). *Acta Chim. Acad. Sci. Hung.*, 1955, **8** (1–3), 1–9.—The complete and rapid reaction, catalysed by $Fe^{\cdots}$, of a CrO_4'' - oxalate complex with methyl orange to give a colourless product forms the basis of a method for determining Cr^{VI}, at concn. as low as 0·001 per cent. and in the presence of large excess of $Fe^{\cdots}$, Al, Ca, Mg, Zn, $Cr^{\cdots}$, $Cu^{\cdot\cdot}$, $Ni^{\cdot\cdot}$, Cd, $Ti^{\cdots\cdot}$, $U^{\cdots\cdots}$, Cl' and PO_4'''. *Procedure*—Add Na_2CO_3 soln. (10 per cent.) (0·1 ml) and $FeCl_3$ soln. (5 per cent.) (0·1 ml) to the CrO_4'' solution (2 ml), dissolve the ppt. in conc. H_3PO_4 (0·5 ml) and add saturated Na oxalate (0·5 ml). Titrate the solution with 0·001 per cent. methyl orange. Cr^{III} can be similarly determined after oxidation with $KMnO_4$. J. P. STERN

1699. **Perchloric acid method for determining chromium in steel.** P. F. Agafonov (Stalin Metallurgical Works, Zlatoust). *Zavod. Lab.*, 1955, **21** (11), 1311–1312.—The presence of H_3PO_4 prevents loss of Cr as volatile compounds in the perchloric acid method for determining Cr in steel. For low-chromium steel, 1 g is dissolved in 20 to 25 ml of dil. HCl (1 + 1) and 3 to 5 ml of conc. HNO_3, the liquid is evaporated to 5 ml, 20 ml of an oxidising mixture (850 ml of 40 per cent. $HClO_4$ and 150 ml of H_3PO_4, sp. gr. 1·7) are added, and the solution is boiled for 6 to 8 min. The cooled solution is mixed with 30 ml of water and 10 ml of 7·5 per cent. NaCl solution and the solution is boiled for 2 or 3 min. to reduce Mn. Water (80 to 100 ml) and 10 to 15 ml of dil. H_2SO_4 (1 + 4) are added and the cooled solution is titrated with a ferrous solution, with phenylanthranilic acid as indicator. For high-chromium steel, 0·2 g is dissolved in 20 ml of conc. HCl and 2 to 3 ml of conc. HNO_3, the solution is evaporated to a vol. of 3 to 5 ml, 20 to 25 ml of an oxidising mixture (925 ml of 40 per cent. $HClO_4$ and 75 ml of H_3PO_4, sp. gr. 1·7) are added and the liquid is boiled until the colour changes from green to dark brown. Boiling is continued for 8 to 10 min., the liquid is cooled, and 25 to 30 ml of water and 10 ml of 7·5 per cent. NaCl solution are added. The solution is boiled to remove chlorine, treated with 20 ml of dil. H_2SO_4 (1 + 4), and diluted to a vol. of 250 to 300 ml. If V is absent, a direct titration is made with a ferrous solution. Otherwise a back-titration is carried out. G. S. SMITH

1700. **Use of perchloric acid for determining chromium in steel.** N. V. Belogorskaya (Novocherkasskiĭ Veterinary Inst.). *Zavod. Lab.*, 1955, **21** (11), 1313.—Loss of some Cr as CrO_2Cl_2 occurs in the normal perchloric acid method for determining Cr in steel. In the method described, volatile compounds of Cr are absorbed in water. The steel (0·2 to 2 g) is heated in a 100-ml conical flask with 30 ml of dil. HCl (1 + 4) to dissolve most of the sample. Perchloric acid (40 per cent.) (2 ml) is added, the flask is connected through a condenser to an absorption vessel containing 50 ml of water, and the contents of the flask are boiled until only a few drops of an orange - red liquid are left. The contents of the absorption vessel are poured into the flask and the liquid is boiled to remove chlorine. An excess of ferrous solution is added and the excess is determined by means of permanganate solution. G. S. SMITH

1701. **Determination of chromium, vanadium and tungsten by activation analysis using low-level neutron sources.** M. Huaringa (Inst. Nuclear Studies, Univ. Liège, Belgium). *Nature*, 1956, **177**, 85.—By the very quick, sensitive method described, Cr, V and W are determined by pptg. them as silver chromate, vanadate and tungstate, respectively, with a neutral soln. of $AgNO_3$ and determining the amount of element present by means of thermal neutron activation of silver. After exposure to a thermal neutron source of 270 mg of Ra-Be for 10 min., Cr and W are not activated, but V is activated fairly well, this activity adding to that of Ag. Up to 25 mg per sq. cm, no corrections for self-absorption are required for either of the three insoluble salts. In determining the unknown concn., the ppt. is collected on a fritted-glass filter, dried and weighed. The total ppt. or a known aliquot part is then collected on a Plexiglas plate and activated. The amount of element on the plate is read from a standard curve, which indicates the activity of the ppt. as a function of the quantity of the metal present. O. M. WHITTON

1702. **Separation and estimation of sexa- and quinque-valent molybdenum by paper chromatography.** M. I. Candela, E. J. Hewitt and H. M. Stevens (Long Ashton Res. Sta., Bristol, England). *Anal. Chim. Acta*, 1956, **14** (1), 66–69.—The quant. separation of Mo^{V} and Mo^{VI} by descending paper-chromatography with a solvent mixture of H_2O (100 ml) - acetic acid (10 ml) - KSCN (10 g) at 20° C is described. The chromatograms are prepared by the pilot-strip method (*J. Chem. Soc.*, 1953, 1338), with a run of ≃20 cm in the eluent; the bands on the pilot-strip are revealed by spraying with dithiol soln. and exposure to HCl fumes. After treatment of the valency-bands on the main strip with 30 ml (warm) of 10 per cent. KSCN in 2 *N* HCl, the molybdenum pentathiocyanate is extracted into ethyl acetate and determined spectrophotometrically. The error is ±3 per cent. for 50 to 100 μg of Mo, and the lower limit of detection is 10 μg of Mo. W. J. BAKER

1703. **Chemistry of molybdenite.** B. S. Khristoforov, L. N. Gvozdeva and Z. M. Arkhipova. *Zapiski Vsesoyuz. Mineralog. Obschchestva*, 1954, **83** (1), 58–60; *Referativnyĭ Zh., Khim.*, 1955, Abstr. No. 21,456.—Samples of 200 to 250-mesh molybdenite containing 50·82 per cent. of Mo, 35·86 per cent. of S and insignificant amounts of pyrite and chalcopyrite are treated with 200 times their wt. of Na_2CO_3 solutions of various concn. In 2 *N* Na_2CO_3 at room temp. the solubility is 0·10 to 0·12 per cent. and in 1·5 to 3 *N* Na_2CO_3 at 90° C the solubility is ≤ 0·5 per cent. In 8 *N* HCl the solubility at room temp. is 0·37 per cent. and at 90° C < 0·47 per cent. The practical insolubility of molybdenite in Na_2CO_3 and HCl solutions can be used for determining molybdenum oxides in molybdenum ores and for phase analysis of ores and concentrates. G. S. SMITH

1704. **New reagents for tungsten. I. Review of existing methods.** J. Zato (Fac. de Ciencias, Salamanca, Spain). *Inf. Quím. Anal.*, 1955, **9** (6), 195–205.—The determination of W by pptn. with benzidine by the method of von Knorre gives low results for W in minerals, but correct results with simple ortho-, meta- and para-tungstates. It was found that alkali silicates or sulphates did not affect the pptn. if it was carried out hot, but that the presence of alkali (Na or K) chlorides caused low results by increasing the solubility of the W - benzidine complex. These chlorides would be present during the analysis of tungsten minerals, after neutralisation of the alkaline fusion mixture with HCl. L. A. O'NEILL

1705. **The detection of sexavalent uranium with salicylaldoxime.** G. Almássy (Debrecen Phys. Res. Inst. Hung. Acad. Sci.). *Magyar Kém. Foly.*, 1955, **61** (12), 404–406.—Many metal ions, even in low concn., interfere with the detection of uranyl ions with salicylaldoxime (**I**). The effect of the interfering ions is masked by EDTA (disodium salt), but this also enters into the uranyl - **I** complex, giving it a reddish-brown colour. *Procedure*—Ignited coal-ash, free of carbon particles (1 g), was warmed in conc. HNO_3 (5 ml) for 10 min. and the soln. was then evaporated to dryness. The residue was dissolved in cold 7 *N* HNO_3 (5 ml) and the basin rinsed with the same solvent (1 ml). The combined soln. were centrifuged, the liquid was extracted with 1·5 vol. of ether by shaking for 5 min. The ethereal layer was evaporated to dryness in a porcelain dish and the dry residue was dissolved in *N* HCl (0·5 ml) and water (1 ml). To this soln., in a test-tube, satd. aq. **I** (0·5 ml) and satd. EDTA (disodium salt) (1 ml) were added, followed after 1 min. by conc. aq. NH_3 (0·5 ml). A reddish-brown colour shows the presence of uranyl ions. The sensitivity is 5 μg per ml. A. G. Peto

1706. **Spectrophotometric determination of uranium.** M. T. Beck and E. Hantos (Univ. Szeged, Hungary). *Acta Chim. Acad. Sci. Hung.*, 1955, **8** (1–3), 233–240.—The stability of the UO_2 - morin complex in aq. ethanol between pH 4 and 7 is confirmed and the ratio of UO_2 to morin is shown to be 1:2. Uranium, as the UO_2 - morin complex, can be determined spectrophotometrically at 430 mμ or colorimetrically, with a Pulfrich photometer with filters S_{42} and S_{47}; the accuracy is within $\pm$10 per cent. Elements of group III of the Periodic Table which interfere should be separated from U by paper chromatography. (*Cf. Anal. Abstr.*, 1955, **2**, 1520.) J. P. Stern

1707. **Colorimetric determination of moderate concentrations of uranium in perchloric acid solutions.** L. Silverman and L. Moudy (Atomics International, N. Amer. Aviation, Inc., Downey, Calif., U.S.A.). *Anal. Chem.*, 1956, **28** (1), 45–47.—Uranium is determined spectrophotometrically in solutions of perchloric acid (up to 65 per cent. by vol.) at 415 to 420 mμ and 25° C. Above 65 per cent., Beer's law is not obeyed, but $Al^{\cdots}$, $Cd^{\cdot\cdot}$, $Pb^{\cdot\cdot}$, $Fe^{\cdots}$, $Th^{\cdots\cdot}$ and $Zr^{\cdots\cdot}$ do not interfere. D. A. Pantony

1708. **Determination of small amounts of uranium in coal.** C. Ujhelyi (Phys. Res. Inst. Hung. Acad. Sci., Debrecen, Hungary). *Magyar Kém. Foly.*, 1955, **61** (12), 437–442.—Details are given of a method wherein the coal-ash is digested with H_2SO_4 or H_2SO_4 and HF. With H_2SO_4 the method is quicker but less accurate. The U^{VI} is then separated by means of ammonium carbonate or ether and is determined colorimetrically with $K_4Fe(CN)_6$. The values obtained agree well with those obtained by radioactive counting. A. G. Peto

1709. **Ultramicro-colorimetric argentimetry.** W. J. Kirsten (Univ. Uppsala, Sweden). *Mikrochim. Acta*, 1955, (5–6), 1086–1087.—Chloride, bromide, iodide and sulphide in a mixture of phosphoric acid, ethyl acetate and chloroform react with the silver of the diphenylthiocarbazone - silver complex. Diphenylthiocarbazone is liberated and can be measured at 598 mμ. The reaction is very sensitive. C. A. Slater

1710. **Inorganic paper-chromatography. XXI. Chromatography of potassium halides.** Shirô Harasawa and Suehiko Hayano (Yamaguchi Univ., Ube, Japan). *J. Chem. Soc. Japan, Pure Chem. Sect.*, 1955, **76** (7), 789–792.—The paper-chromatographic separation of Cl′, Br′ and I′ was studied with developing agents containing ammonia or organic bases, including pyridine, aniline and quinoline. Satisfactory separation is effected by the use of a mixture of quinoline (or pyridine), aq. NH_3 soln. (13 per cent.) and ethanol (10 + 5 + 5), (R_F Cl′, 0·4; Br′, 0·6; I′, 0·8). Potassium ions are adsorbed below the band of Cl′ (R_F, 0·1 to 0·2) and are detected independently of the adsorption bands of the halides. K. Saito

1711. **Separation of trace amounts of fluoride from tervalent aluminium salts by ion exchange.** J. Coursier and J. Saulnier (L'École Supérieure de Phys. et de Chim. de Paris, France). *Anal. Chim. Acta*, 1956, **14** (1), 62–65.—A chromatographic procedure for the quant. separation of $\lessapprox$100 p.p.m. of F′ from $\simeq$100 mg of Al^{III} in alkaline soln. is described. About 50 ml of the soln. of the Al^{III} salt is passed through an anion-exchange column (Amberlite IRA-400 converted to the Na form; 54 cm long by 1 sq. cm section) at 2 to 2·5 litres per min. The $Al^{\cdots}$ are eluted, at the same rate, with 0·2 *N* NaOH (280 to 300 ml); the F′ are then eluted with *N* NaOH (100 to 250 ml) and can be determined colorimetrically by standard methods. The error is $\pm$0·5 to 2 per cent. for 1 to 10 mg of F′ in the sample, but increases for lower concn. of F′. W. J. Baker

1712. **Quantitative determination of fluorine in insoluble fluorides by distillation as boron fluoride.** Z. T. Maksimȳcheva and Sh. T. Talipov. *Trudy Sredneaziat. Univ., No. 55, Khim. Nauk*, 1954, (7), 129–134; *Referativnȳĭ Zh., Khim.*, 1955, Abstr. No. 40,414.—About 2 g of the sample of superphosphate or phosphorite flour containing 12 to 23 mg of F′ are placed together with 1 g of B_2O_3 in a distillation flask fitted with a condenser and dropping funnel and boiled with 100 ml of dil. H_2SO_4 (1 + 2), introduced through the dropping funnel. Air is passed through the liquid at the rate of 10 to 20 bubbles a min., and water vapour and BF_3 are collected in a receiver. When fumes of SO_3 appear in the condenser dil. H_2SO_4 (1 + 3) is added to the contents of the flask. One 200-ml vol. of distillate and then two 100-ml vol. of distillate are collected. Each fraction is neutralised to alizarin red S, evaporated twice, cooled, acidified with 2 per cent. HCl solution, treated with 1 ml of a buffer solution of chloroacetic acid and its sodium salt, and then titrated with thorium nitrate solution to a pink end-point. A correction is necessary for the amount of H_2SO_4 which has distilled over with the BF_3. The titrated distillates are combined, mixed with the ppt., diluted to 500 ml, and filtered. A gravimetric determination of SO_4'' is carried out on an aliquot portion of the filtrate. With the amount of H_2SO_4 so found, a control experiment is carried out. The reaction with thorium nitrate is—

$$4BF_3 + 12H_2O + 3Th(NO_3)_4 = 3ThF_4 + 4H_3BO_3 + 12HNO_3.$$

G. S. Smith

1713. **Properties and analytical applications of the rare-earth fluorides.** G. E. Knudson (State Univ. of Iowa, U.S.A.). *Dissert. Abstr.*, 1955, **15** (10), 1717.—Because rare-earth fluorides are converted into oxyfluorides and oxides in air of normal

humidity and at temp. required for quant. ignition of precipitates, they are useless for the direct gravimetric determination of fluorine. In a relatively simple and rapid method, fluorine is distilled from the sample as fluorosilicic acid, which is precipitated with a known amount of lanthanum nitrate. The excess of lanthanum is then determined gravimetrically by precipitation with cupferron and ignition to the oxide. Better accuracy is attained with this method in the range from 10 mg to 100 mg than with other available methods.
O. M. WHITTON

1714. **Effect of pH on high-salt - thorium fluoride titration.** D. F. Adams and R. K. Koppe (State Coll., Pullman, Wash., U.S.A.). *Anal. Chem.*, 1956, **28** (1), 116–117.—Variations of pH have considerable effect on the precision of determination of F′ by means of a titration with Th⁺⁺⁺⁺. Results are expressed graphically so that corrections for pH variation can be made. It is recommended that a pH of 2·90 should be used in order to minimise the effect.
D. A. PANTONY

1715. **Electrometric titration of chloride in small volumes.** J. A. Ramsay, R. H. J. Brown and P. C. Croghan (Cambridge Univ., England). *J. Exp. Biol.*, 1955, **32** (4), 822–829.—The first of two procedures described is an adaptation for micro quantities of the conventional titration method with 0·01 *N* $AgNO_3$ and electrometric detection of the end-point. The vol. of sample required is 0·2 to 10 μl containing 10 to 500 m-equiv. of Cl′ per litre, which can be determined with a standard deviation of $<\pm 1$ per cent. The apparatus required is described. In the second method, Ag^+ are added to the titrating soln. by passing a current through a silver electrode in series with a condenser. The charge developed in the condenser is linearly related to the amount of Ag^+ liberated, and the charge required to produce a definite potential (240 mV) in the titrated soln. is a measure of the Cl′ present. This method can be used for vol. of sample down to $0{\cdot}5 \times 10^{-3}$ μl, and 10^{-4} μg of Cl′ can be measured with a standard deviation of $<\pm 1$ per cent. Details are given for the construction of the necessary apparatus and electrical circuits.
W. H. C. SHAW

1716. **Colorimetric determination of iodine with Variamine blue [4-amino-4′-methoxydiphenylamine].** L. Erdey and F. Szabadváry (Tech. Univ., Budapest). *Acta Chim. Acad. Sci. Hung.*, 1955, **8** (1–3), 191–201 [in German]; *Magyar Kém. Foly.*, 1955, **61** (11), 341–345.—The action of iodine on Variamine blue (**I**) affords a blue product useful for the colorimetric determination of I (0·5 to 12 μg), preferably at pH 3. *Procedure*—Dilute the weakly acidic soln. (25 to 600 μg of I; $\geq$25 ml) with acetic acid - Na acetate buffer (pH 3), add 2 ml of a 1 per cent. soln. of pure **I** and make up to 50 ml with buffer soln. Shake and set aside the closed flask for 2 min.; then measure (within 10 min.) the extinction of the blue soln. (Pulfrich photometer with S57 filter) against H_2O as blank. If technical **I** is used, a 1 per cent. soln. of **I** should be used as blank. Interference by I′ is dependent on the pH; at pH 3 a 4000-fold excess of I′ does not interfere, but if the excess of I′ is <100-fold, the reaction can be carried out in 0·01 *N* HCl or 0·01 *N* H_2SO_4. All oxidising agents interfere; Zn and Cd form complexes with **I** and more **I** must be used; Pb (>10 mg), Hg (>10 mg), Cu (10 to 100 mg) and Bi (any quantity), CN^-, oxalate, borate, NO_2^- (trace), $PO_4^{\prime\prime\prime}$ (1 mg) and NO_3^- (>100 mg) interfere. Most other ions do not interfere.
J. P. STERN

1717. **A new method of estimation of iodides.** J. V. S. Ramanjaneyulu and B. K. Shukla (Central Salt Research Inst., Bhavnagar). *J. Sci. Ind. Res., B, India*, 1955, **14** (10), 536–537.—The reaction between chromic acid and hydriodic acid has been employed in devising a method for the estimation of iodides in iodised salt. Preliminary investigations are reported. Several estimations with iodide solutions ranging from 0·1 *N* to 0·001 *N* have been carried out and the results obtained with solutions of concn. of about 0·02 *N* are tabulated. Concordant results have been obtained. *Suggested procedure*—Iodised salt (5 to 10 g) is dissolved in about 50 ml of water, and 5 ml of $K_2Cr_2O_7$ solution (0·01 *N*), 3 ml of H_2SO_4 (4 *N*) and 2 ml of sodium oxalate solution (0·1 *N*) are added. The reaction mixture is set aside for about 10 to 15 min. after stoppering the bottle. Then 6 ml of ferrous ammonium sulphate solution (0·1 *N*) are added, followed by a few ml of KI solution (0·1 *N*). The iodine liberated is titrated with standard thiosulphate (0·002 *N*).
G. C. JONES

1718. **Electrometric titration of iodide with dichromate.** M. N. Sastri and C. R. Murty (Andhra University, Waltair, India). *Naturwissenschaften*, 1955, **42** (18), 511–512.—A satisfactory titration is obtained if the acid concentration is kept very low (0·25 *N* HCl or H_2SO_4) and oxalate is added to the iodide solution as a catalyst. The end-point is sharp and coincides with a rise in potential of 80 to 100 mV for 0·1 ml of added dichromate. Under the conditions employed no reaction takes place between oxalic acid and the dichromate, the consumption of the dichromate being stoicheiometrically equivalent to the iodide present.
E. KAWERAU

1719. **Use of iodine chloride in analytical chemistry. I. Determination of iodides, thiosulphates, sulphites and ferrocyanides.** J. Čihalík and D. Vavreinová (Karlovy Univ., Prague, Czechoslovakia). *Chem. Listy*, 1955, **49** (5), 693–699.—The potentiometric determination of I′, $S_2O_3^{\prime\prime}$, $SO_3^{\prime\prime}$ and $Fe(CN)_6^{\prime\prime\prime\prime}$ with 0·1 *N* ICl has been studied. Iodides can be determined in neutral or weakly acidic soln. in concn. as low as 0·0004 *M*, and in the presence of a large excess of Cl′ and Br′. Sulphites are best determined indirectly by titrating the excess of ICl with 0·1 *N* As_2O_3. To prepare a 0·1 *N* soln. of the reagent, treat a soln. of KI (11·068 g) in H_2O (50 ml) with a soln. of KIO_3 (7·134 g) in H_2O (250 ml), add at once conc. HCl (200 ml) free from Fe^{+++} and free Cl, and dilute to 1 litre. The pale-orange soln. has a potential of 750 mV (*vs.* the S.C.E.). Its factor is determined by titration with standard solutions of I′, $S_2O_3^{\prime\prime}$ (indirectly) or $Fe(CN)_6^{\prime\prime\prime\prime}$; it remains practically constant for at least 6 months.
G. GLASER

1720. **Micro-detection and colorimetric micro-determination of manganese (II) by a catalytic reaction.** G. Almássy and I. Dezső (Med. Univ., Debrecen, Hungary). *Acta Chim. Acad. Sci. Hung.*, 1955, **8** (1–3), 11–21.—Manganous ions in H_3PO_4 solution reduce $CrO_4^{\prime\prime}$ at 100° C in the presence of oxalate to green Cr^{+++}; the intensity of the green colour is a function of the concn. of Mn. By this reaction 0·5 μg can be detected and >3 μg can be determined colorimetrically (S47 blue filter) with an accuracy $>\pm 4$ per cent. Iron, Al, Ca, Mg, Cu, Ni, Ti and Zn do not interfere in the determination, which is particularly applicable to Mn in natural waters, plants and food.
J. P. STERN

1721. **Determination of manganese in air.** M. V. Alekseeva and B. A. Gaukhman. *Inform.-metod. Materialy Gos. Nauch. Issledovatel. Sanit. Inst.*, 1954, (5), 26–28; *Referativnyĭ Zh., Khim.*, 1955, Abstr. No. 40,387.—Air at the rate of 1 litre per min. is passed for 30 min. through 4 ml of an absorbing solution prepared by dissolving 10 g of $(NH_4)_2S_2O_8$ in 150 ml of 5 per cent. H_2SO_4 solution, adding 25 ml of H_3PO_4 and 15 ml of 0·1 *N* $AgNO_3$ and diluting to 200 ml. Manganese forms MnO_4'; H_3PO_4 is used to prevent interference from Fe in the subsequent colorimetric determination. The solution is diluted to 5 ml and heated at 100° C for 2 min. It is then cooled and the colour is compared with that of standards prepared from a solution of 23·75 ml of 0·1 *N* $KMnO_4$ and 20 ml of dil. H_2SO_4 (1 + 9), decolorised with 0·3 per cent. H_2O_2 solution and then diluted to 250 ml giving a content of 0·01 mg of Mn per ml. The method is sensitive to 0·0008 mg of Mn in 5 ml of solution. The error is $\pm$0·001 mg.
G. S. Smith

1722. **Potentiometric method for determining large amounts of manganese.** D. A. Pchelintsev (Inst. Geol. Sci., Acad. Sci. SSSR). *Zavod. Lab.*, 1955, **21** (11), 1307–1309.—High concn. of Mn in ores, ferromanganese, etc., can be determined on a semi-micro scale by the potentiometric titration of the solution containing $MnSO_4$ with 0·005 *N* $KMnO_4$, in the presence of sodium pyrophosphate.
G. S. Smith

1723. **Photocolorimetric determination of manganese in ores and minerals.** D. N. Finkel'shteĭn (Central Lab. Ural Geol. Dept.). *Zavod. Lab.*, 1955, **21** (11), 1309–1311.—Manganese ore (0·5 g) in a moist beaker is treated with 5 ml of conc. HCl. The liquid is evaporated to dryness, 15 to 20 ml of dil. H_2SO_4 (1 + 4) are added, the liquid is kept near the b.p. for 10 to 15 min., 5 or 6 drops of 10 per cent. H_2O_2 solution are added, and the solution is evaporated to fuming. The cooled residue is taken up in water, the solution is filtered, and to the filtrate are added 5 ml of dil. H_3PO_4 (1 + 1), containing 50 mg of KIO_4 per litre (the solution is coloured owing to free iodine), and 10 ml of a filtered 0·12 per cent. solution of KIO_4 in 0·1 *N* H_2SO_4. The solution (50 to 60 ml) is heated at $<$90° C for 15 to 20 min. If, after 5 min., no colour has developed, 2 to 3 ml of aq. NH_3 solution (sp. gr. 0·91) are added and heating is continued. The solution is cooled and diluted to 100 ml in a calibrated flask. The extinction is measured after 1 hr., with a green filter. The manganese content is obtained from a calibration curve. The method is applicable to determinations over the range 0·002 to 10 per cent.
G. S. Smith

1724. **Potentiometric determination of iron by X-ray oxidation.** M. A. Dewar and R. R. Hentz (N. Carolina State College, Raleigh, U.S.A.). *Nucleonics*, 1955, **13** (2), 54–56.—Samples (10 ml) containing 10^{-4} to 10^{-3} *M* Fe$^{\cdot\cdot}$, 10^{-3} *M* NaCl and 0·4 *M* H_2SO_4 were irradiated in a beam of X-rays at $\simeq$100 roentgens per min., with continuous stirring. The oxidation - reduction potential slowly increased and reached a limiting value in a time which was directly proportional to the original concentration of Fe$^{\cdot\cdot}$, with a precision of a few per cent.
E. N. Jenkins

1725. **Simultaneous spectrophotometric determination of iron and aluminium with oxine.** Kenji Motojima (Coll. Tech., Ritsumeikan Univ., Kamigyo-ku, Kyoto). *J. Chem. Soc. Japan, Pure Chem. Sect.*, 1955, **76** (8), 903–906.—Both Al (5 to 50 μg) and Fe (5 to 100 μg) oxinates are quant. extracted from an aq. soln. of pH 4·8 to 5·4 with chloroform. The extinction of the $CHCl_3$ layer at 470 mμ is proportional to the amount of Fe and that at 390 mμ to the sum of Fe and Al. A weakly acid soln. ($\simeq$35 ml) is treated with oxine (2 g in 200 ml of water containing 5 ml of glacial acetic acid) and the pH value is adjusted with 2 *M* ammonium acetate. The aq. soln. is extracted with $CHCl_3$ (10 ml); the organic layer is dried with Na_2SO_4 (1 g) and submitted to spectrophotometry. Salts of alkali and alkaline-earth metals (and of Mg) do not interfere.
K. Saito

1726. **Spectrophotometric determination of ferric iron as perchlorate.** Masayoshi Ishibashi, Tsunenobu Shigematsu, Yuroku Yamamoto, Masayuki Tabushi and Toyokichi Kitagawa (Univ., Sakyo-ku, Kyoto, Japan). *J. Chem. Soc. Japan, Pure Chem. Sect.*, 1955, **76** (7), 758–761.—The extinction (at 300 mμ) of an aq. soln. of ferric perchlorate in 3·6 *N* H_2SO_4 is proportional to the amount of Fe (0·05 to 10·0 p.p.m.). The presence of Cu, Ni, Co, Zn, Mn, Al, Na or K does not vitiate the estimation. The method appears to be suitable for the differential colorimetry of a macro amount of Fe. The sample (crude Fe_2O_3) is dissolved in HCl, oxidised with bromine water and heated with $HClO_4$ to dryness. The product is dissolved in 500 ml of water and a 10 to 20-ml portion with H_2SO_4 (1 + 3) (40 ml) is made up to 100 ml. The extinction of the soln. is measured at 300 mμ. A similar soln. containing a known amount of Fe (2·50 to 5·00 mg per 100 ml) is placed in the compensating optical path. The average deviation from the mean is $<$0·2 per cent.
K. Saito

1727. **The effect of phosphoric acid on the titration of ferrous, tervalent titanium and quinquevalent molybdenum ions in the presence of quadrivalent uranium.** Chozo Yoshimura (Kinki Univ., Fuse, Osaka, Japan). *J. Chem. Soc. Japan, Pure Chem. Sect.*, 1955, **76** (8), 883–885.—The oxidation of U^{IV} by ceric sulphate is masked by the addition of H_3PO_4 (1 + 1) (40 ml for $<$10 mg of U) to such an extent that no reaction takes place within several hr. Ferrous iron, Ti$^{\cdots}$ or Mo$^{\cdots\cdots}$ can be titrated with ceric sulphate either potentiometrically (platinum - tungsten electrode), or with ferroin (or diphenylamine) as indicator in the presence of uranous ions. The change in colour at the end-point is not affected by the presence of a large amount of U.
K. Saito

1728. **Polarographic determination of ferrous and ferric iron in refractory minerals.** G. S. Bien and E. D. Goldberg (Scripps Inst. Oceanography, Univ. Calif., La Jolla, Calif., U.S.A.). *Anal. Chem.*, 1956, **28** (1), 97–98.—The mineral is fluxed with $NaBF_4$ under N at 1000° C, and the cake is dissolved in a soln. of citric acid and sodium citrate as complexing agent and KNO_3 as supporting electrolyte for the subsequent polarographic analysis. This is run from +0·15 to −0·35 V to give a composite polarogram, on which the anodic current is proportional to the Fe$^{\cdot\cdot}$ and the cathodic to the Fe$^{\cdot\cdot\cdot}$ concn.
D. A. Pantony

1729. **Determination of non-metallic inclusions in steel by electrolysis and chlorination. Electrolytic determination of carbides in steel.** M. Šícha (VŽKG, Ostrava, Czechoslovakia). *Hutn. List.*, 1954, **9** (1), 2–11.—Inclusions in steel isolated by

electrolysis can be separated from carbides by chlorination *in vacuo*. Sulphides are separated from the electrolytic residue and are determined in a special apparatus, the H_2S being collected in a Cd acetate trap and the CdS determined photometrically. The apparatus for chlorination is described and illustrated. It is capable of working at high temp. (up to 1200° C) and under high vacuum (10^{-2} mm of Hg). Details of the method and of its use are given. Electrolysis is carried out under nitrogen for 24 to 48 hr. in an apparatus (illustrated) in which the anode and cathode compartments are separated by a diaphragm. The anodic electrolyte contains Na citrate (5 per cent.), NaCl (2 per cent.), KBr (1·5 per cent.) and KI (0·1 per cent.), adjusted to pH 3; for high austenitic steels, potassium thiocyanate (2 per cent.) replaces the other salts of K. The electrolyte is replaced at the rate of 1 drop per sec. to prevent evolution of oxygen at the anode. Any insoluble matter crumbling off the electrode is collected on a filter at the outlet of the anodic electrolyte. A. O. JAKUBOVIC

1730. **Gas analysis in steel mills.** R. R. Swain and J. A. Hays (Bailey Meter Co., Cleveland, Ohio, U.S.A.). *Instrum. and Automation*, 1955, **28** (11), 1940–1944.—A review is given of instrumental methods of analysing and controlling the gases involved in steel production. The review deals with blast furnaces, open hearths, soaking pits, reheating furnaces, annealing furnaces, boilers and oxygen plants. G. SKIRROW

1731. **New iodimetric determination of cobalt based on formation of iodopentamminecobalt (III) nitrate.** R. G. Yalman (Antioch College, Yellow Springs, Ohio, U.S.A.). *Anal. Chem.*, 1956, **28** (1), 91–93.—A soln. of Co^{++} is treated with excess of NH_4NO_3 and aq. NH_3 at pH 9 and a known (excess) volume of standard I in KI soln. After pptn. of the iodopentamminocobaltic nitrate, the excess of I is titrated potentiometrically with standard As^{+++}. No interference occurs with Cu^{++}, Ni^{++}, Zn^{++} and Cd^{++}, nor with Al^{+++}, Fe^{+++}, Bi^{+++} and Sn^{++++} in the presence of tartrate. D. A. PANTONY

1732. **Determination of cobalt by anodic electrodeposition. Utilisation of isotope-dilution method.** S. Salyer and T. R. Sweet (Ohio State Univ., Columbus, Ohio, U.S.A.). *Anal. Chem.*, 1956, **28** (1), 61–63.—Cobalt is deposited as hydrated cobaltic oxide at an anode from a solution to which has been added a known wt. of ^{60}Co. After a short time of deposition the deposit is weighed and examined with a Geiger tube and scaler. The cobalt concn. is deduced from the isotopic dilution method. D. A. PANTONY

1733. **The use of ion-exchange resin for the separation of geometrical isomers. I. The separation of *flavo*- and *croceo*-cobalt salts.** Motokichi Mori, Muraji Shibata and Junji Azami (Kanazawa Univ., Kanazawa, Japan). *J. Chem. Soc. Japan, Pure Chem. Sect.*, 1955, **76** (9), 1003–1007.—The separation of *flavo*- and *croceo*-cobalt salts [*cis*- and *trans*-dinitrotetra-amminocobaltic chlorides] was studied by the use of Amberlite IR-120(NH_4). The exchange equilibrium constants were measured by the batch method in 10^{-2} to 10^{-4} *M* soln. The *trans* compound is adsorbed preferentially; the ethylenediamine complexes are adsorbed less preferentially than the corresponding ammine complexes. For the separation of the isomers, elution with 0·5 *M* NH_4Cl (rate of flow, 1 ml per min.) appears to be most suitable. K. SAITO

1734. **Composition of complex compounds of bivalent cobalt with dimethylglyoxime.** L. S. Nadezhina and P. N. Kovalenko (Rostov-on-Don Univ., U.S.S.R.). *Dokl. Akad. Nauk SSSR*, 1955, **101** (6), 1073–1074.—Extinction studies in the aq. systems $CoSO_4$ - H_2Dm (where H_2Dm is dimethylglyoxime) in the presence of sodium stannite and of various amounts of aq. NH_3 or NaOH suggest that the coloured products are $[Co(HDm)_2.2NH_3]$ or $[Co(HDm)_2.2H_2O]$. R. TRUSCOE

1735. **Estimation of small quantities of nickel in the presence of cobalt and iron.** M. Pontet (Inst. Recherches de la Sidérurgie, Saint-Germain-en-Laye, France). *Chim. Anal.*, 1955, **37** (10), 338–339.—A colorimetric estimation is given for small amounts of Ni in alloys that are rich in Co and Fe. Most of the Fe is extracted from HCl soln. with *iso*propyl ether, and the Co is complexed as $[Co(CN)_6]'''$ at a pH >8. The Ni is then converted into the dimethylglyoxime complex, which is extracted in $CHCl_3$. *Procedure*—A soln. of the metals in 10 ml of HCl (sp. gr. 1·19) and 20 ml of H_2O is transferred to a separating funnel with the aid of 30 ml of HCl and then 20 ml of H_2O; 10 ml of HCl are then added, and the soln. is extracted with *iso*propyl ether (3 × 30 ml). The combined aq. layers are neutralised (aq. NH_3 and HCl) and made up to 200 ml. To an aliquot containing >100 to 150 μg of Ni are added 2 drops of aq. NH_3, followed by 10 ml of H_2O, 10 ml of ammonium citrate soln. (50 per cent.), 2 ml of aq. NH_3 and 20 ml of KCN soln. (4 per cent.). The mixture is stirred, then 0·5 ml of H_2O_2 soln. (30 per cent.) is added, and the soln. is boiled for 5 min. When the temp. has fallen to between 60° and 70° C, 2 ml of formaldehyde (35 per cent.) are introduced, followed by 2 ml of aq. NH_3. After keeping at 65° to 70° C for 5 min., the soln. is cooled to 30° C, and then poured into a separating funnel, rinsing out with H_2O (4 × 5 ml). The Ni is then extracted with $CHCl_3$ as the dimethylglyoxime compound and washed three times with 10 ml of aq. NH_3 (1:50). The Ni is recovered by extracting with 0·5 *N* HCl (4 × 10 ml) and to the extract are added, with stirring, 5 ml of 2 *N* HCl, 10 ml of ammonium citrate soln. (50 per cent.), 10 ml of saturated bromine water, 4 ml of dimethylglyoxime reagent and 10 ml of aq. NH_3. The soln. is made up to 100 ml, allowed to stand for 5 min. and its extinction is measured at 540 mμ. A calibration curve is made from standard soln. of Ni. J. H. WATON

1736. **4-Methyl- and 4-*iso*propyl-*cyclo*hexane-1:2-dione dioxime. Gravimetric reagents for nickel and palladium.** C. V. Banks and D. T. Hooker (Iowa State College, Ames, Iowa, U.S.A.). *Anal. Chem.*, 1956, **28** (1), 79–81.—4-Methyl- and 4-*iso*propyl-*cyclo*hexane-1:2-dione dioxime are examined as possible reagents for the determination of nickel and palladium. Precipitation, collection and stoicheiometry are satisfactory and interferences are similar to those of diacetone dioxime. D. A. PANTONY

1737. **Interaction of platinum-group elements with 1:2:3-benzotriazole.** R. F. Wilson and L. E. Wilson (Texas Southern Univ., Houston, Tex., U.S.A.). *Anal. Chem.*, 1956, **28** (1), 93–96.—Palladous chloride is pptd. with 1:2:3-benzotriazole at 60° to 90° C, in the presence of sodium

acetate - acetic acid buffer and sodium ethylenediaminetetra-acetate. The ppt. is stoicheiometric and in a convenient form for weighing. No interference occurs with SO_4'' and NO_3', but moderate amounts of $PtCl_4''$ and $AuCl_4'$ cause interference by co-pptn. Alternatively, the $PdCl_4''$ are titrated amperometrically in dilute soln. with the reagent in the presence of sodium acetate - acetic acid buffer and gelatin. D. A. PANTONY

1738. **A study of electrolytic methods for the separation of platinum, palladium, rhodium and iridium.** J. Bubernak (Ohio State Univ., U.S.A.). *Dissert. Abstr.*, 1955, **15** (6), 955–956.—The Gilchrist - Wichers procedure was modified to include electrolytic methods. After separation of Os and Ru by distillation, Pt is separated from Pd, Rh and Ir by pptn. of the last three metals as their hydrated dioxides; Pt may then be determined in the filtrate by electrolytic deposition. The ppt. of hydrated dioxides is dissolved in HCl and the soln. is adjusted to contain 0·5 *M* HCl and 3·0 *M* NaCl with 8 g of hydrazine dihydrochloride added per 175 ml of soln. Palladium is deposited from this soln. at a cathode potential of +0·050 V *vs.* the S.C.E. at 60° C and Rh at −0·250 to −0·275 V at 60° C. Iridium can then be pptd. as the hydrated dioxide and ignited in H to the metal. N. E.

1739. **3-Hydroxy-1 : 3-diphenyltriazine as reagent for palladium.** N. C. Sogani and S. C. Bhattacharya (National Chem. Lab., Poona, India). *Anal. Chem.*, 1956, **28** (1), 81–83.—3-Hydroxy-1 : 3-diphenyltriazine forms a stoicheiometric insoluble green complex with palladous ions at pH 2 to 3 in the presence of tartrate. In this way Pd can be separated from $PtCl_6''$, and $Ni^{••}$, $Co^{••}$, $Zn^{••}$, $Al^{•••}$, $Cr^{•••}$, $Cd^{••}$, $Mn^{••}$, $Sb^{•••}$, $Bi^{•••}$, $As^{•••}$, $Be^{••}$, $UO_2^{••}$, $Fe^{••}$, $Fe^{•••}$, $Zr^{••••}$, $Ce^{••••}$ and NO_3' do not interfere in the determination of Pd. The reagent is claimed to be superior to dimethylglyoxime. D. A. PANTONY

1740. **Determination of palladium by means of *p*-thiocyanatoaniline.** E. S. Przheval'skiĭ, V. I. Shlenskaya and N. F. Ogarkova. *Vestnik Moskov. Univ.*, 1954, (10), 59–64; *Referativnŷĭ Zh., Khim.*, 1955, Abstr. No. 40,395.—*p*-Thiocyanatoaniline (m.p. 56° C, colourless needles, difficultly sol. in water but easily sol. in organic solvents) gives with $Pd^{••}$ a yellow ppt., $PdCl_2.2NH_2C_6H_4SCN$, decomp. >150° C, insol. in water, acids, alkalis and organic solvents. For quant. determination of Pd the acid solution of pH >0·25 is treated with a 1 per cent. solution of the reagent in ethanol in excess, and the ppt. is collected after 15 min. in a glass filtering crucible, washed with 20 ml of 20 per cent. aq. ethanol, then with water, and dried at 130° to 140° C. The factor is 0·2232. No interference is caused by Fe^{III}, Cu^{II}, Pd^{IV} and Ir^{IV}.
G. S. SMITH

1741. **The analysis of phosphating preparations used for the protection of steel surfaces.** G. H. Bush, D. G. Higgs and F. W. Box (Min. Supply, Armament Res. Dev. Estab., Fort Halstead, Kent, England). *Analyst*, 1955, **80**, 885–896.—Phosphating preparations used for the protection of steel surfaces contain the phosphates of Zn, Mn and Fe, with or without HNO_3, and subsidiary amounts of Cu, Ni, Ca, Cl' and SO_4''. Appropriate chemical methods are described for the determination of these constituents with alternative absorptiometric methods for Cu, Mn and traces of Ni. Nitroguanidine occurred in one solid sample. This is easily separated from the phosphates of Mn and Zn by boiling the sample with water and filtering. After suitable dilution of the filtrate the nitroguanidine is determined spectrophotometrically at 262 mμ. A. O. JONES

1742. **Spectrographic analysis of the getter surface of metallic valves.** A. N. Prokop'eva and I. G. Lazareva. *Sb. rats. predlozheniĭ M-va élektrost. i élektroprom-sti SSSR*, 1954, (33), 18–19; *Referativnŷĭ Zh., Khim.*, 1955, Abstr. No. 21,497.—The amount (0·2 to 3 mg) of sprayed barium on the getter surface can be determined by the following procedure. The spectrograph ISP-65 with glass optical system, 4 amp. a.c. arc, copper electrodes, 0·2 mm slit-width, 1·5 mm arc gap, 285 mm distance between slit and condenser, 80 mm distance between condenser and arc, 20 sec. pre-arcing time, and comparison of the intensity of the line Ba 4554·04 A with that of the background in the region of 4552 A, are the conditions used for spectrographic analysis. The getter surface is washed off with 2 ml of 25 per cent. HCl solution and the solution is diluted to 100 ml. A drop is placed on the heated lower electrode, which is arced for 10 sec. after the drop has dried. This operation is repeated twice but with 5 min. arcing each time. The standard solution contains 0·0005 to 0·01 per cent. of Ba. Fresh electrodes are used for each sample. A calibration curve is constructed from the photometric readings of the spectra of the standards on the plate. A complete qual. analysis of the material investigated is also obtainable from the spectrum. G. S. SMITH

1743. **X-ray determination of quartz and felspar in kaolin.** M. Abbad y Berger and F. Gómez Ruimonte ("Alonso de Santa Cruz" Inst.). *An. Real. Soc. Esp. Fís. Quím.*, 1955, **51**B (12), 671–688.—A quick X-ray method for determining the approx. concn. of felspar and quartz in kaolins, based on the study of equal intensity lines in Debye patterns, is considered. Kaolin - quartz and kaolin - felspar scales are prepared, and the most suitable lines chosen. A similar method is employed for the analysis of kaolins which have been heat-treated at 1300° C for 5 hr.; for this, mullite - quartz and mullite - cristobalite lines have been studied, and the most suitable ones selected. These are tabulated conveniently, with indications of their relative intensity. Nineteen samples of kaolin from different sources have been analysed by this method, and the results checked by chemical analysis. Quartz in kaolin at a concn. of 0·1 per cent., and felspar in a concn. of 2 per cent. can be detected.
C. A. FINCH

1744. **Determination of cement content in plastic concrete.** W. G. Hime and R. A. Willis (Portland Cement Ass., Chicago, Ill., U.S.A.). *Bull. A.S.T.M.*, 1955, No. 209, 37–43.—In the new method described, the cement and fine aggregate are first separated from the sample (≃4 lb) by agitation of successive portions under water in a small container made of 30-mesh brass-screen. The −30-mesh solids are collected, dried and weighed; two 25-g samples are then centrifuged for 11 min. (1200 r.p.m.) in 50-ml tubes containing a mixture of CCl_4 and tetrabromoethane adjusted to sp. gr. 2·80, so that the cement sinks and the aggregate floats. The vol. of the cement layer is measured and the cement content is extrapolated from a calibration curve relating known cement contents and measured vol. for batches containing from 3 to 7 sacks of cement per cu. yd. One

determination takes $\simeq 1\frac{1}{4}$ hr., and the results are accurate to within about one-eighth of a sack per cu. yd. W. J. BAKER

See also Abstracts **1609**, **1894**, **1895**, **1922**, **1925**, **1929**, **1930**, **1931**, **1939**, **1944**.

3.—ORGANIC ANALYSIS

1745. **The rapid micro-combustion procedure.** G. Ingram (Courtaulds Ltd., Berks., England). *Chem. & Ind.*, 1956, (5), 103–107.—The rapid combustion "empty tube" technique of Belcher and Spooner (*J. Chem. Soc.*, 1943, 313) for burning organic compounds is briefly described. In comparison with Pregl's method for the micro-determination of C and H, the principal advantages are the elimination of the tedious combustion procedure, the ease of maintenance and simplicity of the apparatus, and the rapidity with which proficiency may be acquired by an inexperienced operator. Modifications of the procedure for determining halogens and S are reviewed, with emphasis on the methods available for the determination of these elements in the absorbate. The technique has been tentatively applied to the micro-determination of N. A. R. ROGERS

1746. **The direct determination of oxygen (in organic compounds).** A. Hintermaier and R. Grützner (Firma Henkel et Cie, G.m.b.H., Düsseldorf). *Mikrochim. Acta*, 1955, (5–6), 944–949.—Modifications of the Schütze and Unterzaucher method for the direct determination of oxygen in organic substances are described. These include the use of porcelain in place of quartz combustion-tubes and a special purification of the nitrogen used. C. A. SLATER

1747. **Determination of sulphur by wet combustion with perchloric acid.** P. O. Bethge (Swedish Forests Products Res. Lab., Stockholm, Sweden). *Anal. Chem.*, 1956, **28** (1), 119–122.—Sulphur is determined by employing wet combustion, with a nitric - perchloric acid mixture, followed by reduction of the S to H_2S with a reduction mixture. The final determination is iodimetric. In the analysis of 14 pure compounds, recoveries ranged from 98·7 to 101·4 per cent. and the method is applicable over the range 0·01 to 100 per cent. of sulphur. Application to wood and pulp samples was successful. G. P. COOK

1748. **Determination of sulphur in alcohol by combustion.** R. J. Peltonen, P. Neuenschwander and H. Suomalainen (Res. Lab. State Alcohol Monopoly, Helsinki, Finland). *Z. Lebensmitt-Untersuch.*, 1955, **102** (4), 245–253.—A modification of the method used by the American Society for Testing Materials for the determination of S in petroleum products is described; three duplicate determinations can be carried out simultaneously in the apparatus illustrated. *Procedure*—A weighed quantity of alcohol (20 g) is burnt in a special burner with a cotton wick, tests being made on samples containing S in the range 0·015 to 6·0 mg per 100 ml of alcohol. The gases are passed through a glass filter into an ammoniacal soln. containing H_2O_2, any S being converted into $(NH_4)_2SO_4$. Benzidine hydrochloride is added and the sulphate pptd. is washed, dissolved in hot HCl and measured spectrophotometrically at 250 mμ. The method is suitable for the determination of S in methanol and other alcohols. S.C.I. ABSTR.

1749. **Determination of nitrogen by the method of Kirsten.** S. J. Clark and B. Dando (British Nylon Spinners Ltd., Pontypool, Mon.). *Mikrochim. Acta*, 1955, (5–6), 1012–1015.—A modification of apparatus used in the Kirsten method for the determination of nitrogen is described. C. A. SLATER

1750. **Determination of phosphorus in organic phosphates.** I. Ubaldini and F. C. Maitan (Istituto Chim. Ind. Politecnico, Soc. Montecatini, Milan). *Chim. e Ind.*, 1955, **37** (11), 871–873.—For the determination of P in org. phosphates of low volatility, the material is destroyed either by treatment at 215° to 230° C with a mixture (2:3 by vol.) of conc. HNO_3 and $HClO_4$ (70 per cent.), or by calcination with Eschka mixture. After the former procedure, the P in the solution may be determined volumetrically with $AgNO_3$ in the presence of glycine, or gravimetrically as NH_4MgPO_4. After the calcination procedure, the gravimetric method should be used. Results on tritolyl phosphate, OO-diethyl O-*p*-nitrophenyl thiophosphate and lecithin are reported. L. A. O'NEILL

1751. **The micro-determination of chlorine, bromine and iodine in organic compounds.** A. S. Inglis (Liverpool Univ., England). *Mikrochim. Acta*, 1955, (5–6), 934–943.—An improvement in the Parr bomb procedure for the micro-determination of halogens is described. After fusion, the resulting halide is titrated potentiometrically with 0·005 *N* $AgNO_3$, with a platinum indicator electrode and a mercury - mercurous sulphate reference electrode. There is no difficulty in titrating bromide or iodide ions and the electrode system is sensitive to 0·02-ml additions of $AgNO_3$ soln. near the end-point. For the determination of chloride, undesirable materials are separated from the halide with an ion exchanger before the titration. C. A. SLATER

1752. **Periodate - permanganate oxidations. II. Determination of terminal methylene groups.** R. U. Lemieux and E. von Rudloff (Nat. Res. Council of Canada, Prairie Reg. Lab., Saskatoon, Canada). *Canad. J. Chem.*, 1955, **33** (11), 1710–1713.—Terminal methylene groups give formaldehyde in high yield on oxidation with aq. IO_4' and a trace of MnO_4' at pH 7 to 10. Yields range from 12 to 100 per cent. for different compounds, and are considerably higher than those obtained by ozonolysis. Different compounds give max. yields at different pH, but the general procedure recommended is as follows. The compound (0·005 millimole) in 5 ml of H_2O is treated with enough 0·1 *N* K_2CO_3 to give a pH of 7 to 7·6. To this soln. 5 ml of 0·1 *N* KOH, 10 ml of 0·02 *M* $NaIO_4$ and 1 ml of 0·005 *M* $KMnO_4$ are added. After 1 min. 5 ml of 0·1 *N* HCl are added and the soln. is made up to 25 ml. After a further 15 to 60 min. the reaction is stopped by adding 2 ml of *M* $NaAsO_2$ and 2 ml of 2 *N* H_2SO_4. The formaldehyde is then determined with chromotropic acid by the method of Lambert and Neish (*Canad. J. Res.*, 1950, **28**B, 83).

III. Determination of *iso*propylidene groups. E. von Rudloff. *Ibid.*, 1955, **33** (11), 1714–1719.—*iso*Propylidene groups give acetone in nearly quantitative yield (>98 per cent.) on oxidation with IO_4' and a trace of MnO_4', at pH 7·2 to 7·5. Compounds insoluble in water can be dissolved in 25 per cent. dioxan or 25 per cent. pyridine. The material (0·5 millimole) is dissolved in 100 ml of water. This solution (50 ml) is adjusted to pH 7·2

to 7·5 with K_2CO_3, treated with 20 ml of a solution containing 98·33 millimoles of $NaIO_4$ and 1·67 millimoles of $KMnO_4$ per litre, and made up to 100 ml. After 1 hr., 10 ml are withdrawn and treated with 1 ml of *M* $NaAsO_2$, 1 ml of 2 *N* NaOH and 10 to 15 ml of water. The acetone formed can be distilled (a redistillation after acidification is necessary with pyridine) and determined by titration with iodine, or determined colorimetrically with salicylaldehyde. A. B. DENSHAM

1753. **Unsaturation determination by acid-catalysed bromination.** R. E. Byrne, jun., and J. B. Johnson (Carbide and Carbon Chem. Co., S. Charleston, W. Va., U.S.A.). *Anal. Chem.*, 1956, **28** (1), 126–129.—The method of Kaufman, in which unsaturation is determined by using a soln. of Br in methanol saturated with NaBr, was modified to give better reproducibility. Results were generally reproducible to within ±0·3 per cent.
G. P. COOK

1754. **Use of the polarographic method for the analysis of unsaturated organic compounds.** A. V. Ryabov and G. D. Panova. *Doklady Akad. Nauk SSSR*, 1954, **99** (4), 547–549; *Referativnyĭ Zh., Khim.*, 1955, Abstr. No. 40,341.—Unsaturated organic compounds, most of which are not reduced at the dropping-mercury electrode, can be determined polarographically if they are first brominated with a solution of Br in methanol saturated with NaBr. Gaseous compounds are absorbed in 40 ml of the reagent (3 *M* in Br), 5 to 10 per cent. aq. NH_3 is added during cooling to decolorise the solution, a suitable indifferent electrolyte is also added to give a vol. of 100 ml, and a polarogram is obtained at 20° ± 0·2° C with a capillary having $m^{\frac{2}{3}}t^{\frac{1}{6}} = 1$. Liquid organic compounds are determined similarly. The method is applicable to the study of mixtures of organic compounds. G. S. SMITH

1755. **Determination of alpha,beta-unsaturated compounds by reaction with sodium sulphite.** F. E. Critchfield and J. B. Johnson (Carbide and Carbon Chem. Co., S. Charleston, W. Va., U.S.A.). *Anal. Chem.*, 1956, **28** (1), 73–75.—An acidimetric method for the determination of α,β-unsaturated compounds, based on their reaction with $NaHSO_3$, is described. In the reaction a substituted sodium sulphonate is formed with a corresponding decrease in acidity. The reaction conditions for several compounds are listed. Reproducibility is within ±0·2 per cent. and good agreement with other methods is obtained. G. P. COOK

1756. **Determination of alpha,beta-unsaturated compounds by reaction with morpholine.** F. E. Critchfield, G. L. Funk and J. B. Johnson (Carbide and Carbon Chem. Co., S. Charleston, W. Va., U.S.A.). *Anal. Chem.*, 1956, **28** (1), 76–79.—The method is based on the reaction of α,β-unsaturated compounds with morpholine in the presence of acetic acid catalyst. The tertiary amine formed is determined by titration with acid after destruction of excess of morpholine with acetic anhydride. Reproducibility is within ±0·2 per cent. and good agreement with other methods is reached. Fifteen compounds were successfully assayed.
G. P. COOK

1757. **The role of aluminium halogenoalcoholates in the Meerwein - Ponndorf - Verley reduction. III. Determination of carbonyl groups with a modified Meerwein reduction.** I. Simonyi, G. Tokár and G. Gál (United Pharm. and Foodstuff Factory, Budapest). *Magyar Kém. Foly.*, 1955, **61** (11), 367–372.—The reduction gives acetone, which is determined after distillation. The reagent, containing 8 to 10 per cent. of aluminium *iso*propoxide and 4 to 5 per cent. of aluminium "chloro*iso*propoxide" (**I**) is prepared by adding aluminium foil (12 g) to a soln. of $HgCl_2$ (0·1 g) in H_2O- and acetone-free *iso*propyl alcohol (**II**) (500 ml) and heating to boiling point. Dry CCl_4 (10 ml) is added through the condenser (with cooling, if necessary). When the Al has dissolved, the soln. is ready and can be stored for 1 month. After the addition of H_2O, the **I** content can be determined by Volhard's method. The method depends on determining the acetone formed by distillation into aq. hydroxylamine hydrochloride; the acetoxime is then determined by titrating the excess of hydroxylamine hydrochloride. A. G. PETO

1758. **The identification of a chemical species with the microscope.** A. Kofler. *Chim. Anal.*, 1955, **37** (12), 399–408.—The use of the microscope and special apparatus (including a heated microscope stage) for the determination of the physical constants of organic compounds is described. By the techniques given it is possible to identify a very small quantity of material. Eutectic temperatures may be readily determined and used to help in identification. By making use of 24 glass-powders of known refractive indices (from 1·3400 to 1·6877) it is possible to determine the refractive index of a molten solid by comparing the test substance with each of the glasses in turn. C. A. SLATER

1759. **Improved methods of detection in organic spot-tests.** F. Feigl and C. Stark (Lab. Produção Mineral, Min. Agric., Rio de Janeiro). *Mikrochim. Acta*, 1955, (5–6), 996–1003.—Improvements of tests for alcohols, chloral, salicyl alcohol, salicin, sulphoxylated compounds, and some organic reducing agents are described. C. A. SLATER

1760. **Fractionation of hydrocarbons by azeotropic distillation with fluorochemicals.** B. J. Mair (Carnegie Inst. Tech., Pittsburgh, Pa., U.S.A.). *Anal. Chem.*, 1956, **28** (1), 52–56.—The complete resolution of a mixture of branched paraffins and cycloparaffins into the two separate groups can be effected by azeotropic distillation by using fluorochemicals alternately with polar organic compounds as the azeotrope-forming substances.
G. P. COOK

1761. **Fractionation of hydrocarbons by adsorption with added components.** B. J. Mair, M. J. Montjar and F. D. Rossini (Carnegie Inst. Tech., Pittsburgh, Pa., U.S.A.). *Anal. Chem.*, 1956, **28** (1), 56–61.—A method is described for separating hydrocarbons by adsorption, in which the fractionation takes place in the presence of two added components, one in the adsorbed phase and one in the liquid phase. The added components (2-methoxyethanol or diethylene glycol monomethyl ether in the adsorber phase and heptacosafluorotributylamine or "perfluorocyclic ether" in the liquid phase) were particularly effective for the separation of branched paraffins from cycloparaffins.
G. P. COOK

1762. **A rapid chromatographic method for the determination of impurities in ethylene.** N. H. Ray (I.C.I., Alkali Div., Northwich, Cheshire, England). *Analyst*, 1955, **80**, 853–860.—An apparatus is

described and illustrated for the determination of non-olefinic impurities (other than CO_2) in ethylene. The sample is carried by a stream of air-free CO_2, first through a column of active charcoal saturated with Br which removes the ethylene, and then through a column of plain active charcoal by which the ethylene-free sample is fractionated. The fractions are collected in a nitrometer containing KOH soln. and the volumes collected are plotted against the time. The graph indicates the emergence of the successive fractions. Experiments with samples of ethylene containing known amounts of ethane, methane and hydrogen gave accurate and reproducible results. A. O. JONES

1763. **Quantitative determination of ethylene glycol [ethanediol] in water.** E. R. Hess, C. B. Jordan and H. K. Ross (Paint and Chem. Lab. Div., Aberdeen Proving Ground, Md., U.S.A.). *Anal. Chem.*, 1956, **28** (1), 134–135.—The procedure is based on the periodate scission of vicinal glycols followed by the formation of the insol. $AgIO_3$. The time of appearance of the $AgIO_3$ ppt. is proportional to the concn. of ethanediol and, under critical conditions, samples containing $\leqslant 0{\cdot}10$ per cent. of ethanediol are determined with $< \pm 2{\cdot}0$ per cent. error. G. P. COOK

1764. **The determination of the propionyl index according to Pesez. An application to the assay of poly(ethylene glycol).** A. Sezerat (Services de Recherches Roussel-Uclaf, Paris-Romainville). *Ann. Pharm. Franç.*, 1955, **13** (7–8), 516–519.—The method of Pesez for the determination of hydroxyl groups with propionic anhydride (*Anal. Abstr.*, 1925, **2**, 935) is applied to poly(ethylene glycol) having mol. wt. of between 200 and 4000. A comparison is given with a determination by the phthaloyl index. E. J. H. BIRCH

1765. **The lipids of fish. VII. Phosphate esters in the lipids of haddock and cod flesh. [Determination of glycerol.]** J. Olley (D.S.I.R., Torry Res. Sta., Aberdeen, Scotland). *Biochem. J.*, 1956, **62** (1), 107–114.—A micro-chromatographic method is described for the determination of 6 to 20 μg of glycerol. It is based on that of Fisher *et al.* (*Nature*, 1948, **161**, 764). The chromatograms are run in *n*-propanol - water (4:1, by vol.), and the papers are dipped in the $AgNO_3$ - NaOH reagent of Trevelyan *et al.* (*Brit. Abstr. C*, 1951, 260, 410) and then washed with 6 *N* aq. NH_3 soln. to remove substances with the same R_F as glycerol. The method of acid - periodate oxidation is not specific for glycerophosphate esters. J. N. ASHLEY

1766. **The determination of cellulose.** M. Gilly (Maison Sanders, Juvisy, France). *Anal. Chim. Acta*, 1956, **14** (1), 53–56.—The procedures used by different laboratories adopting the original Weende method for the determination of cellulose (*Landwirtsch. Vers.-Sta.*, 1933, **115**, 37) have been examined in respect of their accuracy and reproducibility. A further modification, now used by the Laboratories of the Ministry of Agriculture (Spain), is described in detail. In this method the extensive washings after the successive digestions of the sample with 1·25 per cent. H_2SO_4 and 2·5 per cent. NaOH, respectively, are replaced by neutralisations with alkali and acid of the same strength, thus saving time and limiting sharply the extent of attack on the sample. The soln. is filtered through an Alundum crucible, the residue is washed with boiling H_2O, dried at 103° C for 15 hr. and weighed (w_1). It is then ignited at exactly 700° C for 2 hr. in a thermostatically controlled electric furnace and weighed (w_2) when cool. The cellulose percentage = 100 ($w_1 - w_2$). Results are more reproducible and generally slightly higher than the method involving washings. W. J. BAKER

1767. **The cellulose-dissolving power of the tri-(ethylenediamine) - zinc hydroxide complex.** G. Jayme and K. Neuschäffer (Inst. f. Cellulosechem. Tech. Hochschule, Darmstadt, Germany). *Naturwissenschaften*, 1955, **42** (19), 536.—A very reactive chloride-containing zinc hydroxide is prepared by adding ammonia to a 10 per cent. soln. of $ZnCl_2$ at 90° C. The reaction product is washed, suspended in acetone and finally dried in air. Analysis shows it to be composed of 51 per cent. of Zn and 8·3 to 12·6 per cent. of chloride. The amine - zinc complex is prepared by saturating a 40 per cent. v/v soln. of ethylenediamine with this zinc hydroxide, keeping the temp. at 0° C. The resulting tri(ethylenediamine) - zinc complex should contain at least 6 per cent. of Zn. A soln. containing 8·02 per cent. of Zn is capable of dissolving cellulose to a concn. of 2·8 per cent. These soln. of cellulose are colourless and clear when pure cellulose is used. Commercial cellulose products give rise to solutions of varying turbidity and colour, and the solvent can be usefully employed for grading celluloses of different origin. E. KAWERAU

1768. **Characterisation of starch and related polysaccharides by differential thermal analysis.** H. Morita (Chem. Div., Canada Dept. Agric., Ottawa, Canada). *Anal. Chem.*, 1956, **28** (1), 64–67.—Differential thermal analysis was investigated as a means of characterising starches which showed thermal properties dependent on the method of preparation. Distinct variations were found among amylose and amylopectin fractions. The analysis was extended to glycogen, cellulose and dextran. G. P. COOK

1769. **Studies in starch structure. I. Analytical methods.** C. H. F. Fuller, L. H. Lampitt and L. Coton (Lyons Lab., Hammersmith Rd., London). *J. Sci. Food Agric.*, 1955, **6** (11), 656–660.—A scheme for the examination of a starch fraction is described. The starch fraction (700 mg), preferably in the form of wet amylose - butanol complex or wet amylopectin ppt., is stirred with boiling water (60 ml) in a centrifuge tube, N being passed through the liquor at the same time. On complete dissolution, the soln. is diluted to 70 ml and centrifuged or filtered through a sintered-glass disc giving a soln. of $\simeq 1$ per cent. Duplicate portions of 10 ml are transferred to boiling-tubes (6 in. × 1 in.) and used for the determination of reducing power. With the same pipette, 30 ml of the soln. are added to 10 ml of 0·5 *N* KOH contained in a 50-ml calibrated flask. The alkaline soln. is cooled, made up to the mark, saturated with N and stored at f.p. This soln. is used for the determination of polysaccharide concentration, absorption spectrum of the iodine complex, iodine absorption and limiting viscosity no.

II. Determination of iodine absorption by amperometric titration. L. Coton, L. H. Lampitt and C. H. F. Fuller. *Ibid.*, 1955, **6** (11), 660–664.—The titration cell and electrical circuit are described and illustrated; the important factors in design are listed, including control of temp., prevention of loss of I, exclusion of light, stirring, a suitable electrode system and accuracy of the galvanometer. The

absorption of I by suspensions of starch granules was examined by this method. The absorption is characteristic of the starch species, but is increased if damaged granules are present. Wheat flours have an absorption curve similar to that of the extracted starch: the method can therefore be applied directly to the detection of overmilling in flours. S.C.I. Abstr.

1770. **Determination of aldehydes by mercurimetric oxidation.** J. E. Ruch and J. B. Johnson (Carbide and Carbon Chem. Co., S. Charleston, W. Va., U.S.A.). *Anal. Chem.*, 1956, **28** (1), 69–71.—The method is based on the oxidation of aldehydes to acids by Hg^{++}, which are then reduced to free Hg. The Hg is determined iodimetrically. Application to 12 different aldehydes gave results reproducible to within 0·5 per cent. and close agreement with the hydroxylamine hydrochloride method was obtained. G. P. Cook

1771. **Separation of 2:4-dinitrophenylhydrazones of aldehydes and ketones by paper chromatography.** W. S. Lynn, jun., L. A. Steele and E. Staple (Univ. of Pa., Philadelphia, Pa., U.S.A.). *Anal. Chem.*, 1956, **28** (1), 132–133.—Good separation of the 2:4-dinitrophenylhydrazones of aldehydes and ketones was obtained with phenoxyethanol-impregnated paper as the stationary phase and heptane as the mobile phase. G. P. Cook

1772. **Paper-chromatographic method for the determination of some lower organic acids.** J. Janicki, A. Niewiarowicz and M. Skorupski (Faculty Agric. Tech., Poznan, Poland). *Przem. Chem.*, 1954, **10** (8), 417–420.—Lactic (**I**), acetic (**II**), propionic (**III**) and butyric (**IV**) acids in admixture in the form of their sodium salts are determined by descending paper chromatography on Whatman No. 1 paper, with *n*-butanol - 1·5 *N* aq. NH_3 (1:1), for 60 hours. The best developer was found to be 0·1 per cent. bromocresol purple in 20 per cent. ethanol soln. at a pH of 7·5 (obtained with NaOH soln.), and the paper was dried at 30° C for 45 min. Qualitatively down to 10 μg of **II**, **III** or **IV**, and 5 μg of **I**, can be detected. For quantitative work, 20 to 140 μg of the acid should be present, when the errors of the determinations are about 14 per cent. for **I**, 7 per cent. for **II** and 6 per cent. for **III** and **IV**. The relationship between concn. and the area of the spots is probably linear. The method is quicker and simpler, but less accurate, than column chromatography when applied to biological materials. A. O. Jakubovic

1773. **Chromatography of fatty acid anilides, and related problems.** A. P. de Jonge (Unilever Res. Lab., Vlaardingen, Holland). *Chem. Weekbl.*, 1956, **52** (3), 37–43.—The possibility of employing partition chromatography for the analysis of fatty acids in the form of their anilides is considered. Methods of preparing these derivatives are given. With ascending column separation, *cyclo*hexane was in all cases chosen as the mobile phase while 80 per cent. methanol, 50 per cent. ethanol and 20 per cent. *n*-propanol served as the stationary phase for the ranges C_2 to C_{10}, C_2 to C_8 and C_1 to C_6, respectively. A table of R_F values of the anilides of C_1 to C_{10} is given. Spots are revealed by u.v. light and the absorption spectra can be measured by the method of Tennent *et al.* (*Anal. Chem.*, 1951, **23**, 1748). P. Haas

1774. **Identification of some α-dicarbonyl compounds in the form of quinoxaline derivatives by paper chromatography.** A. Harjanne (Univ. Helsinki, Finland). *Suomen Kem., B*, 1955, **28**, 37–40.—Quinoxaline derivatives of a number of dicarbonyl compounds have been prepared by the action of *o*-phenylenediamine (*cf.* Morrison, *J. Amer. Chem. Soc.*, 1954, **76**, 4483). The amount of the parent dicarbonyl compound used was 1 millimolé except with 2-oxogluconate when 0·1 millimole was used. The reaction mixtures were left overnight in a refrigerator. The ppt. were filtered off and dried in a vacuum-desiccator over silica gel. Paper-chromatographic separation was effected in a chromatographic chamber, with descending techniques and, for solvent, the organic phase of a mixture of equal parts of *n*-butanol and 2 *N* aq. NH_3. The compounds were revealed by a contact copying technique, with short-wave u.v. light, the chromatogram being pressed against a photographic paper; the distance between the lamp and the paper was 55 cm and the exposure was 10 sec. Some R_F values obtained are tabulated. The min. detectable amount of material is 10 μg. P. Haas

1775. **Investigation of colour reaction between *p*-dimethylaminobenzaldehyde and urea or ureido acids.** R. E. Cline and R. M. Fink (Univ. of Calif. Sch. of Med., Los Angeles, Calif., U.S.A.). *Anal. Chem.*, 1956, **28** (1), 47–52.—Studies were made of the colour reaction between *p*-dimethylaminobenzaldehyde and urea or ureido acids with specific regard to its use in paper chromatography. Colour development is suppressed by dilution and by HCl concn. above or below the optimum level. Recommendations are made to achieve max. efficiency for routine use. G. P. Cook

1776. **Polarographic determination of maleic hydrazide.** Tsugio Takeuchi, Naruhito Yokouchi and Kiyoshi Onodo (Yamanashi Univ., Kofu, Japan). *J. Electrochem. Soc. Japan*, 1955, **23** (10), 541–544.—It was found that maleic hydrazide (**I**) ($\simeq 10^{-3}$ *M*) shows one polarographic wave at $-2{\cdot}19$ V *vs.* the S.C.E. in 0·1 *M* LiCl soln. in the presence of an excess of diethanolamine (**II**). (In the absence of **II**, **I** gives a complicated polarogram, consisting of four waves.) The wave height remains unchanged for 48 hr. and is proportional to the concn. of **I** when the amount of **II** is > 6 times the equiv. of **I**. This method is suitable for the determination of the diethanolamine salt of **I** (an agricultural chemical). *Procedure*—The sample (0·5 g) is dissolved in water (100 ml) containing aq. **II** soln. (30 per cent.) (4 ml). A 5-ml portion with *M* LiCl (5 ml) is made up to 50 ml, bubbled with a current of H for 5 min. and submitted to polarography. K. Saito

1777. **Paper-chromatographic separation of ammonium chloride, hydrazine, hydroxylamine, phenylhydrazine and phenylhydroxylamine.** F. H. Pollard and A. J. Banister (Univ. Bristol, England). *Anal. Chim. Acta*, 1956, **14** (1), 70–73.—A descending paper-chromatographic separation of NH_4Cl, hydrazine, hydroxylamine, phenylhydrazine and phenylhydroxylamine, in 0·1 to 0·5 *M* aq. solution, can be made with the solvent system ether - methanol - water - conc. HCl (20:30:15:4 by vol.) at 18° ± 1° C, on acid-washed Whatman No. 1 paper and a run of 20 to 30 cm. Ascending elution is preferred for small amounts or simple mixtures. Provided operating conditions are standardised, the R_F values

are reproducible to ±0·02. Hydrazine, hydroxylamine and their deriv. are revealed by a spray of 0·5 per cent. w/v picryl chloride in ethanol, followed by drying and exposure to NH_3. Double spots, depending on the distance of the starting line from the solvent level, are described. W. J. BAKER

1778. **Coulometric determination of organic bases in acetonitrile [methyl cyanide].** C. A. Streuli (American Cyanamid Co., Stamford, Conn., U.S.A.). *Anal. Chem.*, 1956, **28** (1), 130–132.—Weak organic bases (amines) can be determined coulometrically by using methyl cyanide containing 0·05 *N* $LiClO_4.3H_2O$ as the solvent. Hydrogen ions are produced by anodic oxidation of the water content of this solvent and are detected by the glass - calomel electrode system. Pyridine, diphenylguanidine, triethylamine and benzylamine were determined in milligram quantities, the usual error being $<\pm 2{\cdot}0$ per cent. The method is not applicable to aromatic amines. G. P. COOK

1779. **Detection of oxamide, guanidine and guanidine derivatives in spot-test analysis.** F. Feigl and C. Costa Neto (Lab. Produção Mineral, Min. Agric., Rio de Janeiro). *Mikrochim. Acta*, 1955, (5–6), 969–978.—These compounds are detected by their ability to yield dicyanogen, either by heating with P_2O_5 (oxamide) or by pyrolysis at 220° to 250° C (guanidine and its derivatives). The spot test for the dicyanogen is based on its reaction with 8-hydroxyquinoline in the presence of KCN to give a red coloration. C. A. SLATER

1780. **The action of morpholine on some quaternary ammonium salts and its application to their analysis.** A. Barber, C. C. T. Chinnick and P. A. Lincoln [Milton Industrial Chemicals (London), Ltd., 42–46 Weymouth St., London]. *J. Appl. Chem.*, 1955, **5** (11), 594–599.—A method of analysing quaternary ammonium compounds is based on their reaction with morpholine, when the alkyl group splits off and a tertiary amine and N-alkylmorpholine are formed; the reaction products are then fractionally distilled. In an example, a mixture (100 g) of equal parts of lauryltrimethylammonium bromide and myristyltrimethylammonium bromide was refluxed with morpholine (100 g) for 16 hr. A soln. of NaOH (20 g) in H_2O (500 ml) was added to the cooled product, the oily layer was extracted with four 100-ml portions of C_6H_6, and the mixed extracts were washed with H_2O (50 ml). The soln. was distilled at atmospheric pressure until the vapour temp. was 126° C, thus removing H_2O, C_6H_6 and methylmorpholine. The residue was then fractionally distilled under vacuum. O. M. WHITTON

1781. **Volumetric determination of thiourea.** F. Hernández Gutiérrez (Inst. Alonso Barba, Barcelona). *An. Real. Soc. Esp. Fís. Quím.*, 1955, **51**B (11), 639–641.—The sample (>2 mg of thiourea) is treated with mercuric potassium iodide reagent (0·1 *M*, with 0·13 moles of NaOH per litre) to form a complex, which is boiled under reflux with KI (2 g) for 10 minutes. The ppt. of HgS is centrifuged, washed with aq. KNO_3 (5 per cent.), treated with 2 ml of HNO_3 (6·1 *N*) for 5 min. at 80° to 90° C, centrifuged, washed as before, and oxidised with Br solution (2·6 ml of Br and 10 g of KBr in 20 ml). The excess of Br is then either determined iodimetrically, or removed, and the HBr formed in the oxidation is determined by titration with alkali (0·02 *N*). Satisfactory results are obtained on mixtures of thiourea with ascorbic acid. L. A. O'NEILL

1782. **New method for analysis of dialkylbenzene isomers.** Yu. G. Mamedaliev, I. G. Izmailzade, Sh. Mirzoyeva, T. Zeinalova and Kh. M. Abdullayeva (Petroleum Inst., Azerbaijan SSR). *Dokl. Akad. Nauk SSSR*, 1955, **102** (3), 529–530.—The identity and content of isomeric phthalic acids formed by the oxidation of dialkylbenzene mixtures are derived from measurements of the position and intensity of X-ray diffraction rings given by the powder; the experimental error is about 5 per cent. R. TRUSCOE

1783. **The separation of alkyl aromatic homologues by reversed-phase partition chromatography.** P. R. Enslin and D. E. A. Rivett (Council Sci. Ind. Res., Pretoria, S. Africa). *Chem. & Ind.*, 1956, (1), 23–24.—The columns were packed with non-wetting kieselguhr impregnated with purified *iso*heptane (8 ml in 9 g) and eluted with aq. ethanolic solutions of increasing alcoholic strength. A soln. of the substances (2 to 20 mg of each component) in *iso*heptane (1 ml) was shaken with a degassed suspension of kieselguhr (1 g) in a little aq. ethanol and poured on to the top of the column. The eluates were examined in a u.v. spectrophotometer and their extinctions measured. Separations of a mixture of benzene, naphthalene and phenanthrene and of a dehydrogenation mixture obtained from the bitter principle, cucurbitacin A, were effected, but those of anthracene and chrysene and of the two isomeric methylnaphthalenes were not possible. O. M. WHITTON

1784. **Bromatometric estimation of phenolic carbonyl compounds.** V. V. Ramanujam (Dept. Chem., Univ., Annamalainagar, India). *J. Sci. Ind. Res., B, India*, 1955, **14** (11), 564–566.—Hydroxyacetophenones, 2:4-dihydroxybenzaldehyde and 2:4-dihydroxybenzoic acid are estimated by back-titration after brominating with a large excess of bromate - bromide solution. The error varies from 0 to 0·8 per cent. Quantities of 15 to 40 mg of β-resacetophenone are estimated by direct titration, with methyl orange as indicator. I. JONES

1785. **Colour reaction of salicylic acid and nitrite with cupric ion.** A. L. Underwood (Emory Univ., Ga., U.S.A.). *Anal. Chem.*, 1956, **28** (1), 41–44.—Strict control of variables in the determination of Cu^{++} by the colour reaction with salicylic acid and NO_2' gives greater efficiency. The standard deviation is ≃0·04 p.p.m. and the concn. range is 2 to 9 p.p.m. under the conditions described. G. P. COOK

1786. **Paper chromatography of 2:4-dinitrophenylhydrazones.** E. Kulonen. *Suomen Kem., B*, 1955, **28** (2), 105–106.—Aromatic keto-acid hydrazones can be resolved with mixtures of light petroleum (boiling range 180° to 200° C) and *iso*propyl alcohol (70:30) saturated with water. The R_F values depend on the amount of *iso*propyl alcohol. Aliphatic keto-acid hydrazones migrate very slowly. Neutral hydrazones, such as those of acetone, formaldehyde, acetaldehyde, diacetyl and glycollaldehyde, were separated by liquid paraffin with the addition of some of the above-mentioned light petroleum to decrease the viscosity. The usual length of migration on Whatman paper was 10 cm. Viewed against a white background in

u.v. light, 0·1 μg of hydrazone can be detected. The R_F values increase with the addition of light petroleum; actual values of the hydrazones mentioned are given for a mixture of paraffin and light petroleum in equal parts. 2:4-Dinitrophenylhydrazine and acid 2:4-dinitrophenylhydrazones show no migration. P. HAAS

1787. **Chromatographic resolution of natural coumarins.** A. Chatterjee and A. Choudhury (Univ. Coll. Sci., Calcutta, India). *Naturwissenschaften*, 1955, **42** (19), 535–536.—Acid-washed crude plant extract is chromatographed on alumina columns, with the solvent (ether, light petroleum, benzene) as developer and eluent. The early fractions contain essential oils, glycerides, sterols and triterpenes; the coumarins appear when benzene or a mixture of benzene and ethyl acetate is used in various proportions. For quant. separation, re-chromatographing is essential. Phenolic coumarins adsorb very strongly and can be removed satisfactorily only after extrusion of the column and refluxing of the zone material with methanol or ethanol. Coumarins having alcoholic hydroxyl groups are also strongly adsorbed. E. KAWERAU

1788. **Assay of picric acid by coulometry at controlled potential.** L. Meites and T. Meites (Yale Univ., New Haven, Conn., U.S.A.). *Anal. Chem.*, 1956, **28** (1), 103–106.—Picric acid is quant. reduced from HCl soln. at a mercury cathode, whose potential is controlled at −0·40 V *vs.* the S.C.E., to 2:4:6-triaminophenol. Integration of the current flow during electrolysis in conjunction with a conventional alkalimetric procedure provides the basis for assay of picric acid samples.
G. P. COOK

1789. **Titrations of bases with diphenyl phosphate in some aqueous and non-aqueous solvents.** M. M. Davis and H. B. Hetzer. *J. Res. Nat. Bur. Stand.*, 1955, **54** (6), 309–320.—Various bases, *e.g.*, 1:3-diphenylguanidine, di-*o*-tolylguanidine, guanidine acetate, tribenzylamine, triethylamine, tri-(hydroxymethyl)methylamine, have been titrated with diphenyl phosphate (**I**) in benzene, methanol, H_2O and mixtures of these solvents, in some cases potentiometrically (with the glass - S.C.E. system) and in other cases with indicator dyes. These included Bromophthalein magenta E (tetrabromophenolphthalein ethyl ester), 5-phenyl-9-diethyl Nile blue, the anhydro base of tamarack green (**II**), the anhydro base of Victoria blue B (**III**), phenolphthalein, *p*-naphtholbenzein, sodium salts of sulphonephthaleins, α-Naphthyl red and *p*-ethoxychrysoidine (**IV**). Details for the preparation of **II**, **III** and **IV** are given. Although **I** is shown to be a strong acid, it cannot replace perchloric acid in titrations in acetic acid. Benzoic acid and **I** show different behaviour when the solvent is changed from H_2O to methanol. By using a "non-levelling" solvent in place of H_2O, separate, sharp end-points can be obtained in titrating a mixture of two such acids. O. M. WHITTON

1790. **Paper-chromatographic separation and identification of benzidine and its isomers. XII. Identification of organic compounds.** J. Gasparič, J. Petránek and M. Večeřa (VÚOS, Pardubice-Rybitví, Czechoslovakia). *Mikrochim. Acta*, 1955, (5–6), 1026–1030.—For the separation of benzidine and its isomers, formed during the rearrangement of hydrazobenzene, sheets of Whatman No. 4 paper are treated with a 10 per cent. solution of formamide in ethanol and allowed to dry in air for about 30 min. The chromatogram is developed with *cyclo*hexane for about 2 hr. The spots are identified either by treatment with *p*-dimethylaminobenzaldehyde [1 per cent. solution in ethanol (95 ml) and conc. HCl (5 ml)] or by diazotising and coupling with N-1-naphthylethylenediamine. The diazotisation is carried out by spraying the paper with 5 per cent. HCl, hanging it over a dish of acidified $NaNO_2$ solution for 10 min., and then in the air for a further 15 min. The paper is then sprayed with a 0·5 per cent. soln. of N-1-naphthylethylenediamine dihydrochloride. R_F values and colour reactions are given for hydrazobenzene and five isomers of benzidine. C. A. SLATER

1791. **Polarographic determination of nicotinic and *iso*nicotinic acids and their amides in mixtures.** W. Kemula and Y. Chodowski (Dept. Inorg. Chem., Univ., Warsaw). *Roczn. Chem.*, 1955, **29** (2–3), 839–848.—Polarographic determinations of: (*a*) *iso*nicotinic acid in a mixture with nicotinic acid, (*b*) nicotinic acid in the presence of *iso*nicotinic acid, (*c*) the amides of both acids in the presence of each other, (*d*) *iso*nicotinic acid and its amide from a mixture of both and (*e*) nicotinic acid and its amide in the presence of each other, are described and the results are shown in tables and graphs. It was found that *iso*nicotinic acid can be determined with an error of −7 to +5·4 per cent. in a buffer solution of pH 7·96, after applying a correction (the *iso*nicotinic wave rises by ≃10 per cent. in the presence of nicotinic acid); nicotinic acid can be determined only qualitatively in mixtures with *iso*nicotinic acid. The determination of the amides in the presence of each other at pH 12 gave satisfactory results. Acids and their corresponding amides in mixtures were determined by measuring the amide wave at pH 12 and the sum of acid plus amide wave at pH 8·7 and calculating the acid contents from the difference; the determination of *iso*nicotinic acid and its amide is fairly satisfactory but that of nicotinic acid is less so.
S.C.I. ABSTR.

1792. **Separation of indole compounds by circular chromatography.** H. C. Chakrabortty and S. P. Sen (Bose Inst., Calcutta, India). *Naturwissenschaften*, 1955, **42** (18), 512.—Circles of Whatman No. 1 paper are used. A number of solvent systems were investigated and best results were obtained with *iso*propyl alcohol - aq. NH_3 - water (10:1:1) and *tert.*-butyl alcohol - aq. NH_3 (9:1), the latter mixture being much slower moving. The substances were detected on the paper by spraying with Salkowski's reagent or by ultra-violet fluorescence. R_F values for indolylacetic acid, indolylpropionic acid, indolylbutyric acid, indolylacetamide and N-acetylindoxyl are given. E. KAWERAU

1793. **Determination of vapour - liquid ratio of motor gasoline. Sunbury apparatus.** R. L. Gilpin and F. W. H. Matthews (British Petroleum Co., Ltd., Sunbury-on-Thames, Mdx., England). *Anal. Chem.*, 1956, **28** (1), 99–103.—A measured volume of gasoline at 0° C is introduced into a vapour burette and the volume of vapour in equilibrium with the liquid, at a given temp. and 760 mm pressure, is measured. The repeatability is ±0·5° F at vapour - liquid ratios between 10 and 40.
G. P. COOK

1794. **Determination of tetraethyl-lead in motor spirit by flame photometer.** W. Meine (Esso A.-G., Hamburg-Harburg, Germany). *Erdöl u. Kohle*, 1955, **8** (10), 711–712.—The sample is burned in a flame fed with O and H at 293 and 14 mm pressure (Hg), respectively. The flame is measured, with a monochromator at the Pb line 406 mμ, by means of a photocell, which has been calibrated with fuels of known Pb content. The calibration curve is a straight line; the blank due to flame background emission varies slightly for different types of spirit, benzole giving a higher value than those of cracked and straight-run spirits. The readings can be taken in 1 min. Results compare closely with those by the $KClO_3$ - HNO_3 extraction method.

A. R. PEARSON

1795. **Spectrochemical investigation of the working behaviour of mineral oils. I. Emission spectrographic analysis of inorganic constituents in motor oils.** H. Luther and G. Bergmann (Tech. Hochsch., Brunswick, Germany). *Erdöl u. Kohle*, 1955, **8** (5), 298–304.—Determinations of the inorganic constituents of fresh and used oils are made spectrographically, a rotating electrode being used. In most cases, cadmium palmitate is used as an internal standard. Results of the analyses for the elements Cd, Cu, Zn, Ca, Al, Pb, Cr, Fe and P are examined statistically. J. H. WATON

1796. **Infra-red analysis of bituminous coals and other carbonaceous materials.** R. A. Friedel and J. A. Queiser (Bureau of Mines, Region V, Bruceton, Pa., U.S.A.). *Anal. Chem.*, 1956, **28** (1), 22–30.—The i.r. spectra of coal and coal products, including residues from the vacuum-distillation of coal, have been compared, both liquid-paraffin mulls and the potassium bromide pellet technique being used. Experimental details are described, and several new structural assignments are given for bands in the spectra of high-volatile bituminous coal. Kaolinite is identified as the principal source of a band hitherto assigned to aromatic ethers, and other mineral interferences have been recognised. P. T. BEALE

1797. **Some improvements in the pressed disc technique for the estimation of certain components of pitches and bitumens by infra-red absorption.** G. A. Monnot and P. Morel (Centre d'Études et Recherches des Charbonnages de France, Verneuil, France). *Applied Spectroscopy*, 1955, **9** (4), 180–183.—Samples are mixed with ground KBr and finely ground before being pressed into clear rigid discs. Details of techniques are given and a method of recrystallisation-grinding with a solvent is described which gives greatly improved reproducibility in the spectra of the discs. Examples of the application of the method to the determination of anthracene, carbazole, naphthalene and phenanthrene in tar distillates are given, and it is suggested that the technique could be successfully applied to biological samples. P. T. BEALE

1798. **Chromatography of dyestuffs intermediates. III. Identification and separation of anthraquinonesulphonic acids by paper chromatography.** M. Večeřa, J. Gasparič and J. Borecký (Výzkumný ústav org. synthes, Pardubice-Rybitví, Czechoslovakia). *Chem. Listy*, 1955, **49** (5), 706–708.—The mono- and di-sulphonic acids of anthraquinone were readily separated and identified by paper chromatography on Whatman No. 1 or No. 4 paper, the solvent systems *n*-butanol - aq. NH_3 - water (2:1:1) (system *A*) or *n*-butanol - pyridine - water (3:1:1) (system *B*) being used. The spots were detected by their fluorescence in u.v. light. The following are, respectively, the position(s) of the $-SO_3H$ group(s) in the anthraquinone nucleus, the R_F values of the corresponding acids in the solvent systems *A* and *B* at 21° C, and the colour of their u.v. fluorescence: 1, 0·65, 0·57, yellow; 2, 0·78, 0·72, blue - green; 1:5, 0·04, 0·07, yellow; 1:6, 0·09, 0·15, yellow - green; 1:7, 0·14, 0·19, yellow - green; 1:8, 0·26, 0·35, yellow; 2:6, 0·19, 0·29, green; 2:7, 0·19, 0·29, green. It follows that anthraquinone-2:6- and -2:7-disulphonic acids cannot be separated from each other. The method is a convenient means for determining the purity of commercial preparations and for following the course of sulphonation processes. G. GLASER

1799. **The determination of lignin in flax fibre.** J. F. Couchman (Commonwealth Sci. Ind. Res. Org., Melbourne). *J. Text. Inst.*, 1955, **46** (12), T735–T741.—The effect of the concentration of H_2SO_4, and of time and temp. of the digestion, on the determination of lignin in flax fibre was ascertained. For both wax-free and alkali-extracted samples, digestion for 2 hr. at 20° C with 20 ml of H_2SO_4 (71·8 to 72·0 per cent. w/w, sp. gr. 1·638 to 1·640 at 15° C) was satisfactory when the fibre was completely dispersed in the acid during digestion.

O. M. WHITTON

1800. **The measurement and analysis of the irregularity of jute yarn.** B. L. Banerjee and M. K. Sen. *J. Text. Inst. Proc.*, 1955, **46** (11), 742–755.—Fundamental causes of irregularity of jute yarn are given and results are presented of its measurement as obtained from the variations of diameter and wt. per unit length. The instruments used, namely the photo-electric irregularity tester and the Fielden - Walker evenness recorder and integrator, are illustrated and their calibration is described. The results are compared with those obtained by the direct method of weighing. A suitable procedure for the purpose of routine control is formulated.

O. M. WHITTON

1801. **Paper-chromatographic examination of wool - polyamide fibre blends.** H. H. Bühler and H. Zahn (Chem. Inst., Univ. Heidelberg, Germany). *Melliand Textilber.*, 1955, **36** (10), 980–981.—The paper-chromatographic analysis of polyamides and polyurethanes (*Ibid.*, 1954, **35**, 162) is applied to mixtures of wool, nylon, Perlon and Rilsan fibres. The 10 per cent. aq. solutions of hydrolysates from 100 mg of material are subjected to two-dimensional elution, with *sec.*-butyl alcohol - formic acid - water (75:15:10) in one, and phenol - water (80:20) in the other direction. Ninhydrin solution is used as spray reagent and the chromatograms are dried for 5 to 10 min., at 80° to 100° C. Under the standard conditions of hydrolysis (24 hr. in 6 *N* HCl at 105° C) Rilsan does not completely disintegrate and the hydrolysates of polyaminoundecanoic acid are sparingly soluble in water. The simultaneous chromatography of amino acids of wool as control substances is therefore recommended. R_F values for hydrolysis products of nylon, Perlon, Rilsan and several amino acids of wool are given. Their accuracy is within 2 per cent. for nylon and Perlon, 10 per cent. for Rilsan and 4 per cent. for wool. S.C.I. ABSTR.

1802. **Electrochromatographic separation of cationic surface agents.** S. Fumasoni, E. Mariani and G. Torraca (Ist. Chim. Appl. Ind., Univ. Rome).

Chem. & Ind., 1956, (3), 69–70.—The electrophoretic separation of cationic surface-active agents of formula $\left[\bigcirc\!\!\!\!\bigcirc\!\!\!\!\bigcirc\!\!\!\!

1810. **Analytical technique for silicone polishes.** L. Ivanovszky. *Paint Manuf.*, 1955, **25** (8), 313–317.—Two schemes are outlined for the analysis of water-free polishes. The procedures involve determination of solvent by steam-distillation and of abrasives by filtration after treatment with solvents. Waxes may then be determined by feezing the filtrate, and silicones by evaporation of the dewaxed filtrate; alternatively, silicones may be mechanically separated from waxes after evaporation of the total solvent-soluble material (the silicones separate out in the form of an "eye"). Another scheme is given for silicone emulsion-polishes. Tests for alkalis and emulsifying agents are included. L. A. O'NEILL

See also Abstracts 1897, 1898, 1924, 1928.

4.—BIOCHEMISTRY
INCLUDING DRUGS, FOOD, SANITATION, AGRICULTURE

Blood, Bile, Urine, etc.

1811. **Spectrographic analysis of biological samples. I. The preparation of samples for analysis.** I. L. Smith, E. Yeager, N. Kaufman, F. Hovorka and T. D. Kinney (Western Reserve Univ., Cleveland, Ohio, U.S.A.). *Applied Spectroscopy*, 1955, **9** (4), 167–170.—Biological specimens may be analysed directly by feeding them into a d.c. arc, but more quantitative data are obtained by a preliminary treatment. The techniques of direct analysis, drying, and wet or dry ashing are described, and the advantages and disadvantages of each are discussed. P. T. BEALE

1812. **Indirect colorimetric determination of potassium in blood serum.** H. Bultasová and E. Konopásek (Biochem. labor. Thomayerovy nemocnice, Prague, Czechoslovakia). *Chem. Listy*, 1955, **49** (5), 769–770.—A simple and rapid method for the determination of K in blood serum consists in precipitating K as $K_2NaCo(NO_2)_6$ and in determining the Co colorimetrically with EDTA, according to Přibil and Malik (*Chem. Listy*, 1951, **45**, 237). *Procedure*—The serum (0·4 ml) is mixed with cobaltinitrite reagent (1·5 ml); after 45 min., H_2O (3 ml) is added and the mixture is centrifuged for 10 min. The liquid is decanted, the ppt. is carefully covered with H_2O (3 ml), the mixture is again centrifuged and decanted. The ppt. is suspended in 70 per cent. ethanol (5 ml), the suspension is centrifuged for 10 min. and decanted. The residue is treated with a 5 per cent. soln. of EDTA (disodium salt) (1 ml) and 3 per cent. H_2O_2 (4 ml), and the centrifuge tube is immersed in boiling water until the ppt. dissolves. The intensity of the pink coloration of the cooled soln. is measured and the content of K is read off from a calibration curve prepared from standard solutions of K_2SO_4 (0·2, 0·3, 0·4, 0·5 and 0·6 ml). Serum older than 24 hr. must be deproteinised first by centrifuging a mixture of the serum (1 ml) with 20 per cent. trichloroacetic acid (1 ml); 1 ml of the supernatant liquor is used for the determination. G. GLASER

1813. **The estimation of trace amounts of barium or strontium in biological material by activation analysis.** G. E. Harrison and W. H. A. Raymond (M.R.C. Radiobiological Research Unit, Atomic Energy Res. Estab., Harwell, England). *J. Nuclear Energy*, 1955, **1**, 290–298.—Ashed faecal specimens or ashed residues obtained from a preliminary oxalate pptn. of urine specimens are irradiated for 2 hr. at a slow neutron flux of 2×10^{12} neutrons per sq. cm per sec., together with standards. Barium or strontium (1 to 50-μg amounts) present in the specimens is then measured by gamma-counting the 1·4-hr. ^{139}Ba or the 2·7-hr. ^{87m}Sr produced during the irradiation. Before this measurement, the irradiated samples are dissolved, milligram amounts of inactive carriers are added, the barium and strontium are pptd. as the nitrates with 19 *N* HNO_3, and redissolved in water. Traces of rare earths are removed by co-pptn. with $Fe(OH)_3$ and the barium is isolated by a pptn. of $BaCrO_4$, the strontium remaining in the supernatant liquid. Both barium and strontium are recovered quantitatively, mixed with some residual calcium which does not interfere in the gamma assay. The method is accurate to within 5 per cent. at the levels cited. E. N. JENKINS

1814. **Correction of erratic reagent blanks in determination of serum protein-bound iodine.** D. Sanshuk and L. Aconsky (Walter Reed Army Med. Center, Washington, D.C., U.S.A.). *J. Lab. Clin. Med.*, 1955, **46** (6), 902–904.—High and erratic blank values in the ceric - arsenite method of Barker *et al.* (*J. Clin. Invest.*, 1951, **30**, 55) are traced to the I present as impurity in the NaCl or HCl used as reagents. By dissolving the ashed sample in 3·6 ml of 7 *N* H_2SO_4 and 5·4 ml of water instead of 3 ml of 7 *N* H_2SO_4, 2 ml of 2 *N* HCl and 4 ml of water, blank values of <3 μg of I per cent. are obtainable. W. H. C. SHAW

1815. **A simple micro-method for blood carbon monoxide determination.** H. I. Chinn, N. E. R. Pawel and R. F. Redmond (U.S.A.F. School of Aviation Med., Randolph Field, Tex., U.S.A.). *J. Lab. Clin. Med.*, 1955, **46** (6), 905–909.—In the simple rapid method described the CO liberated from 0·2 ml of blood by treatment with potassium ferricyanide is aspirated slowly through a commercially available (in U.S.A.) indicating tube for the detection of traces of CO in air. The colour change produced in the sample tube is compared with standards prepared from blood containing known amounts of CO or, less accurately, with a calibrated colour chart. The method is suitable for field use and gives results which agree within 10 per cent. of those obtained with the manometric Van Slyke method. W. H. C. SHAW

1816. **Methods for the determination of muscle chloride.** W. O. Caster, R. MacDonald and W. D. Armstrong (Univ. Minnesota, Minneapolis, U.S.A.). *J. Lab. Clin. Med.*, 1955, **46** (6), 910–913.—From a study of the available procedures it is concluded that digestion of the dried defatted muscle with HNO_3 and excess of 0·1 *N* $AgNO_3$ ("open Carius") and titration of the excess of Ag^+ with KSCN is the simplest and easiest method. Alkaline dry-ash methods give satisfactory results provided the samples are thoroughly wetted with at least 2 moles of alkali [NaOH, KOH, $Ca(OH)_2$ or $Ba(OH)_2$] per kg of dry muscle before ashing. W. H. C. SHAW

1817. **The determination of glucose by the Hagedorn and Jensen method.** E. Paschke (Tech. Hochschule, Hanover). *Mikrochim. Acta*, 1955, (5–6), 983–986.—It has been found that, even for high concentrations of glucose, the glucose concentration is strictly proportional to the amount of potassium ferricyanide reduced. C. A. SLATER

1818. **A spectrophotometric method for the determination of lactic acid in samples of fresh blood by a diffusion technique.** H. J. Stark (Göttingen Univ., Germany). *Klin. Wochschr.*, 1956, **34** (5–6), 153–155.—The lactic acid is oxidised by ceric sulphate to acetaldehyde, which is collected in thiosemicarbazide solution in a special diffusion apparatus (*cf. Klin. Wochschr.*, 1955, **33**, 82); the thiosemicarbazone formed is determined by measuring the extinction at 261·5 mμ. Ethanol and acetone interfere and are removed by a preliminary evaporation at 50° C; other substances present in blood do not interfere. From 5 to 50 mg of lactic acid per cent. can be determined with an accuracy of about ±3 per cent. P. S. Stross

1819. **The estimation of small quantities of glycyrrhetinic acid in urine.** V. M. van Katwijk and L. G. Huis in't Veld (Nat. Inst. Public Health, Utrecht, Holland). *Rec. Trav. Chim. Pays-Bas*, 1955, **74** (7), 889–896.—A soln. of glycyrrhetinic acid (10 μg per ml) in 96 per cent. ethanol gives a max. absorption in u.v. at 248 mμ which follows Beer's law. As little as 20 mg of the acid can be extracted from a 24-hr. sample of urine by using a column of Floridin earth (10 cm long × 15 mm in diameter) and eluting with mixtures of benzene and ethanol. A sample of 400 ml taken from a 24-hr. portion of urine is, after hydrolysis with HCl, extracted with $CHCl_3$; the resulting extract is evaporated and then dissolved in 10 or 20 ml of 96 per cent. ethanol. An aliquot of this soln. is evaporated and the residue is dissolved in benzene and transferred to the chromatographic column, which is then eluted with several portions of solvent. A 5-ml aliquot of each eluate is carefully evaporated, the residue is dissolved in 96 per cent. ethanol and its extinction is measured at 248 mμ. No glycyrrhetinic acid could be demonstrated in the 24-hr. urine of patients who had received 1·33, 2·28 or 2·5 g of glycerrhetinic acid per 24 hr., showing that <2 per cent. of the quantity administered is excreted as such or in the form of a salt or easily saponifiable ester. P. Haas

1820. **Spot-test detection of inositol.** F. Feigl and V. Gentil (Lab. Produção Mineral, Min. Agric., Rio de Janeiro). *Mikrochim. Acta*, 1955, (5–6), 1004–1007.—The aq. test solution (1 drop) is evaporated to dryness with HNO_3 (sp. gr. 1·4) (1 drop). The residue is treated with $CaCl_2$ soln. (5 per cent.) (1 drop) and aq. NH_3 (sp. gr. 0·9) (1 drop). The whole is then heated to 180° C, when a red or red-brown colour develops in the presence of inositol. The identification limit is 2·5 μg and limit of dilution 1 in 20,000. C. A. Slater

1821. **Complexing of ATP [adenosine triphosphate] with molybdate.** J. J. Blum and R. W. Chambers (Naval Med. Res. Inst., Bethesda, Md., U.S.A.). *Biochim. Biophys. Acta*, 1955, **18** (4), 601.—The influence of KCl and adenosine triphosphate on the colorimetric determination of PO_4''' by the method of Fiske and Subbarow (*J. Biol. Chem.*, 1925, **66**, 375) is investigated and the precautions necessary to attain correct results are discussed.
W. H. C. Shaw

1822. **Some new solvent systems for the paper chromatography of nucleic acid degradation products.** K. S. Kirby (Royal Cancer Hospital, London). *Biochim. Biophys. Acta*, 1955, **18** (4), 575–576.—For separating the hydrolysis products of deoxyribonucleic acid (**I**) and ribonucleic acid (**II**) on Whatman No. 4 paper by ascending chromatography for 16 hr., the recommended solvent systems are—(*i*) methanol - conc. HCl - water (70:20:10) for **I**, (*ii*) methanol - ethanol - conc. HCl - water (50:25:6:19) for quantitative estimation of the hydrolysis products of **I** and **II** by drying, elution of the spots and subsequent u.v. absorption measurements and (*iii*) ethyl methyl ketone - *tert.*-butyl alcohol - glacial acetic acid - water - conc. HCl (22·5:22·5:35:19:1) for **II**. R_F values for 13 bases, nucleotides and nucleosides in the three solvents are listed.
W. H. C. Shaw

1823. **A study of the conditions and mechanism of the diphenylamine reaction for the colorimetric estimation of deoxyribonucleic acid.** K. Burton (Dept. Biochem., Univ. of Oxford, England). *Biochem. J.*, 1956, **62** (2), 315–323.—The colour reaction of Dische (*Brit. Abstr. A*, 1930, 632) given by deoxyribonucleic acid in the presence of diphenylamine and H_2SO_4 has been studied and modified. The reaction is carried out in the presence of acetaldehyde, and the mixture is kept at 30° C for several hours instead of at 100° C for 3 to 10 min. The modified reaction is more sensitive and specific and less susceptible to interference by other compounds. The mechanism of the reaction is discussed; probably neither free deoxyribonucleic acid nor free ω-hydroxylaevulaldehyde are intermediates. J. N. Ashley

1824. **Rapid method for simultaneous determination of ammonia and carbon dioxide evolved by ninhydrin from amino acids.** P. Linko (Biochem. Inst., Helsinki, Finland). *Suomen Kem., B*, 1955, **28**, 96–99.—A method is described for determining, in the same sample, both NH_3 and CO_2 evolved by the action of ninhydrin on amino acids. The apparatus employed is a slightly modified form of that described by Van Slyke *et al.* (*J. Biol. Chem.*, 1941, **141**, 671). It consists of an inverted U-tube, to either limb of which is attached a small conical flask; the top of the tube is connected to a stop-cock (*C*). From 1 to 10 mg of substance is weighed directly into one of the flasks (*B*) and dissolved in 5 ml of CO_2-free water; about 100 mg of buffer (2·06 g of Na citrate dihydrate and 19·15 g of citric acid) and 100 mg of ninhydrin are added to the soln. and the flask is immediately stoppered. The second flask (*A*) is then freed from CO_2 by passing 250 ml of CO_2-free air through it and 2 ml of $\simeq$0·125 *N* $Ba(OH)_2$ are added. The apparatus is immediately evacuated, shaken vigorously and dipped into a boiling-water bath. After 20 min. the CO_2 is transferred from *B* to *A* by surrounding *A* with ice - water. The apparatus is shaken during this transference and after 2 min. it is cooled and CO_2-free air is introduced through the stop-cock *C*; the liberated CO_2 is then titrated with 0·04 *N* HCl. The contents of flask *B* are treated with six drops of 30 per cent. H_2O_2 and shaken in a boiling-water bath for 3 min., after which the NH_3 is determined by the Parnas - Wagner apparatus. The method has proved to be very rapid and accurate, but alanine, cystine and cysteine give only 90, 50 and 50 per cent., respectively, of the theoretical amount of ammonia. P. Haas

1825. **Positive ninhydrin reaction on paper chromatograms with reducing substances in the presence of traces of ammonia.** W. Klinger (F. Schiller Univ., Jena). *Naturwissenschaften*, 1955, **42** (24), 645.—The reduction product of ninhydrin couples with NH_3 of the laboratory atmosphere and

with further ninhydrin to yield the characteristic pigment. In the presence of the ordinary laboratory atmosphere as little as 50 μg of a monosaccharide could be detected, but if the laboratory air was enriched with ammonia vapour as little as 5 μg gave a colour. Hence sugars on paper chromatograms can simulate amino acids and can falsely intensify amino-acid spots, giving rise to erroneous quant. results. E. KAWERAU

1826. **The paper chromatography of metal complexes of amino acids. (Preliminary communication.)** M. Beck and J. Császár (Inst. Inorg. and Anal. Chem., Univ., Szeged, Hungary). *Magyar Kém. Foly.*, 1955, **61** (12), 447–448.—The interference between amino acids is eliminated by converting them into metal complexes, particularly with Cu^{++}, by shaking them, in aq. soln., with the carbonate. The ascending method was used, on Schleicher and Schüll 2043*a* paper, in aq. dioxan. The paper was sprayed with 0·2 per cent. ethanolic rubeanic acid, followed by ninhydrin. The R_F values of some cupric complexes are—lysine, 0·03; arginine, 0·07; glycine, 0·22; glutamic acid, 0·31; alanine, 0·36; and proline, 0·41. A. G. PETO

1827. **The quantitative determination of amino-acid hydroxyamides.** R. S. Schweet (Calif. Inst. Tech., Pasadena, Calif., U.S.A.). *Biochim. Biophys. Acta*, 1955, **18** (4), 566.—The colorimetric method described gives rectilinear calibration with 0·2 to 1·0 μmole per ml of amino acids. *Procedure*—To 3 ml of reaction mixture (or standard soln.) at pH 7·0 containing neutralised hydroxylamine (1·0 *M* final concn.) add 1·4 ml of 10 per cent. trichloroacetic acid (adjusted to pH 0·9 with NaOH) and 0·6 ml of 2·0 *M* aq. ferric chloride. Centrifuge or filter to remove any protein ppt. and read the colour at 520 mμ after 5 to 20 min. against a blank treated similarly. W. H. C. SHAW

1828. **Determination of leucine, *iso*leucine and phenylalanine in proteins by paper chromatography.** S. Lissitzky and G. Laurent (Faculté de Médecine, 92 rue Auguste-Blanqui, Marseilles). *Bull. Soc. Chim. Biol.*, 1955, **37** (11), 1177–1182.—The protein used was globin prepared from haemoglobin, both adult and foetal, by Anson and Mirsky's method (*J. Gen. Physiol.*, 1930, **13**, 469). For hydrolysis, 20 mg were dissolved in 6 ml of 6 *N* HCl and heated in an evacuated sealed Pyrex-glass tube for 6 hr. at 120° C; the HCl was then removed by distillation under reduced pressure at 60° to 70° C. The resulting amino acids leucine, *iso*leucine and phenylalanine were separated by descending chromatography on Whatman No. 1 or 4 paper, with Work's solvent (*Biochim. Biophys. Acta*, 1949, **3**, 400) of *tert.*-amyl alcohol saturated with water in an atmosphere of diethylamine. Migration was allowed to proceed for 5 days at 15° to 18° C; the paper was then dried at room temp. for 6 to 8 hr. and developed by dipping in a freshly prepared 0·2 per cent. soln. of ninhydrin in pure acetone containing 1 per cent. of acetic acid. After being left in the dark for 15 to 16 hr., the stains were cut out and treated in a test-tube with a 0·1 per cent. soln. of $CdCl_2$ in aq. 60 per cent. methanol. The coloured complex thus formed by the cadmium salt was used for spectrophotometric measurement and comparison aganst a standard. The accuracy of the method is within $\pm$5 per cent. P. HAAS

1829. **The determination of lanthionine in amino-acid mixtures: the separation of its diastereoisomers on columns of ion-exchange resins.** S. Blackburn and G. R. Lee (Wool Ind. Res. Ass., Torridon, Leeds, England). *Analyst*, 1955, **80**, 875–879.—A method for the direct determination of lanthionine in protein hydrolysates or mixtures of amino acids has been developed. Chromatographic separation of the amino acids is effected on columns of the Na form of Dowex 50 by the method of Moore *et al.* (*Brit. Abstr. C*, 1952, 62), the development of the column being limited to the use of a buffer soln. of pH 3·42 but without thiodiglycol. The lanthionine peak emerges between the peaks of serine and glutamic acid but tends to overlap them. Fractions (1 ml) of the eluate are examined with a ninhydrin reagent (prep. described) at pH 1; at this pH overlapping amino acids do not interfere. The extinction of the fractions is measured at 455 mμ and the total amount of lanthionine is ascertained from a calibration graph. The emergence of lanthionine is indicated by the appearance of a yellow double peak followed, after an interval, by a red peak due to proline. The separation of the individual diastereoisomers of lanthionine on these columns and their i.r. spectra are described. A. O. JONES

1830. **Determination of sodium glutamate in protein hydrolysates.** Y. de Hemptinne and W. Vergult. *Fermentatio*, 1955, (5), 225–228.—A protein hydrolysate fraction (2 g), containing impure Na glutamate, is dissolved in water and made up to 500 ml. The solution is chromatographed, first through Amberlite IRC-50 to remove Na^+ and substitute H^+, and then through Amberlite IR-4B, which selectively adsorbs glutamic and aspartic acids. The column is eluted with 500 ml of 2 per cent. Na_2CO_3 soln. A 100-ml aliquot of the eluate, which should have a pH of $\simeq$8·0, is acidified with H_2SO_4 to a pH of 1 to 2, the soln. is heated to boiling point, an excess of $CuCO_3$ powder is added and the whole is filtered hot. The filtrate is carefully evaporated on a steam bath and the residue dissolved in a minimum amount of water. The Cu aspartate is then pptd. by adding $\simeq$40 ml of methanol, filtered off and washed with methanol. The filtrate is evaporated to dryness on a steam bath. The residue is re-dissolved in water, heated to 60° C, and excess of Cu is pptd. with a stream of H_2S. The CuS is filtered off and washed, adding the washings to the filtrate which is then titrated, by acidifying with HCl to pH $\simeq$2, adjusting the pH to exactly 7·0 with 0·1 *N* NaOH, then adding $\simeq$15 ml of formaldehyde (of pH exactly 7·0), shaking and allowing to stand for several minutes, then finally titrating with 0·1 *N* NaOH to a pH of exactly 8·9. S.C.I. ABSTR.

1831. **Quantitative determination of L-aspartic acid by aspartase.** N. Ellfolk (Biochem. Inst., Helsinki, Finland). *Suomen Kem., B*, 1955, **28**, 52–54.—A study has been made of methods for increasing the accuracy of the method of Virtanen and Laine (*Nature*, 1935, **136**, 756; *Biochem. J.*, 1939, **33**, 412) for the determination of aspartic acid based on the reversible reaction with aspartase—

$$\text{L-aspartic acid} \rightleftharpoons \text{fumaric acid} + NH_3.$$

When the enzyme prep. used contain fumarase the equilibrium of fumarase is also established—

$$\text{fumaric acid} + H_2O \rightleftharpoons \text{malic acid}.$$

Hence the deamination of aspartic acid is affected by the fumarase action. Since aspartic acid is not deaminated quant. by aspartase, equilibrium being established corresponding to 50 to 80 per cent. of

aspartic acid, all factors effecting this establishment influence the accuracy of the method. The author has found previously (*Acta Chem. Scand.*, 1953, **7**, 1155) that compounds containing acid groups, such as –COOH, $-SO_3H$, etc., are strong inhibitors of aspartase; accordingly this enzyme cannot be used for the determination of aspartic acid in crude plant extracts containing oxalic, citric or fumaric acids, or diphosphates; these inhibitors must therefore be removed before reliable determinations can be made. The equilibrium can also be affected by other amino acids although these are not real inhibitors; this applies particularly to high concn. of glycine; neutral salts, notably KCl, also have an inhibiting action (see also Trudinger, *Australian J. Exp. Biol. Med. Sci.*, 1953, **31**, 319). The enzyme was prepared by extraction of dried *Propionibacterium petresdonii* powder. P. HAAS

1832. **Quantitative separation and identification of quaternary ammonium bases.** S. Friedman, J. E. McFarlane, P. K. Bhattacharyya and G. Fraenkel (Illinois Univ., Urbana, Ill., U.S.A.). *Arch. Biochem. Biophys.*, 1955, **59** (2), 484–490.—Separation of carnitine, betaine, trimethylamine oxide, and creatine plus creatinine is achieved on columns of prepared Dowex-50 resin washed with *N* HCl. Resin (20 g) in a column (8 mm in diameter) can be loaded with up to 50 mg of the mixed bases and the columns developed at room temp. or in a jacketed (refluxing methanol) column at 65° C. Chromatographic identification of the individual bases in each fraction is by the ascending method on Whatman No. 1 paper developed with ethyl methyl ketone - *iso*propyl alcohol - 0·1 *N* HCl (30:50:20) or *iso*-propyl alcohol - pyridine - water (50:25:25). After development and air-drying, the paper is sprayed with a mixture (1:1) of satd. ethanolic methyl orange and Congo red solutions, redried and treated alternately with NH_3 and HCl vapours. Recovery of the five bases from the resin is 90 to 100 per cent. Choline cannot be eluted from the columns and must be removed by preliminary treatment on Amberlite 1RC-50 resin buffered with sodium acetate - acetic acid at pH 7·0. W. H. C. SHAW

1833. **Paper chromatography of cholesterol and cholesteryl esters.** Č. Michelec (Central Biochem. Lab. Univ. Hospital, Prague). *Naturwissenschaften*, 1955, **42** (18), 509–510.—Ascending chromatography is employed on Whatman No. 3 paper impregnated with a 10 per cent. solution of liquid paraffin in benzene. Acetic acid is used for development. The spots are detected by spraying the paper either with 10 per cent. tungstophosphoric acid in ethanol or 50 per cent. $SbCl_3$ in acetic acid. R_F values for cholesterol and five of its esters are quoted. E. KAWERAU

1834. **Characteristic infra-red absorption bands of steroids with reduced ring *A*. 3-Keto-5α- and -5β-compounds.** H. Rosenkrantz and P. Skogstrom (Worcester Foundation Exp. Biol., Shrewsbury, Mass., U.S.A.). *Anal. Chem.*, 1956, **28** (1), 31–34.—The absorption spectra of twenty-seven 3-keto reduced ring *A* steroids, 12 related structures and 11 acetylated derivatives have been investigated. Bands are assigned to 3-keto-5α- and -5β-compounds and to the 3-hydroxy-5α- and -5β-structures, which permit ready differentiation. Both groups are distinguished from steroids with conjugated ketones by the absence of absorption near 1666 cm^{-1}. In this way the intermediate (3-keto-5α- and -5β-) and terminal (3-hydroxy-5α- and -5β-) reduced compounds can be identified when the α- and β-unsaturated steroids are subjected to chemical or enzymic reduction. P. T. BEALE

1835. **Chemical estimation of progesterone in the blood of cattle, sheep and goats.** J. I. Raeside and C. W. Turner (Univ. Missouri, Columbia, Mo., U.S.A.). *J. Dairy Sci.*, 1955, **38** (12), 1334–1343.—The method given is based on solvent extraction of the sample, partition between organic solvents, separation by paper partition chromatography, elution with ethanol and determination of progesterone by u.v. absorption. A study is made of progesterone levels in normal animal blood and of the levels resulting from the administration of progesterone. W. H. C. SHAW

1836. **Identification of the serum lipids on the electropherogram by differential elution technique.** C. Wunderly (Med. Universitätsklinik, Zürich, Switzerland). *Naturwissenschaften*, 1955, **42** (21), 579.—Electrophoretic separation is carried out in barbitone buffer by the usual method in duplicate on paper strips 8 cm wide. After drying, one of the paper strips is put in a bath of acetone saturated with $MgCl_2$ for 24 hours at −24° C. Phospholipids remain on the paper after this treatment. The same strip is now cut lengthways into two, and one half is treated in a covered bath with light petroleum at ≃30° C for 8 hours, when the light petroleum is renewed and the washing continued for another 16 hours. The treatment removes glycerophosphatides and leaves sphingolipids on the paper. Finally the three strips are stained in the same bath with Sudan black. Neutral fat, esters of fatty acids and chylomicrons are acetone-soluble and therefore do not appear on strips that have been treated with this solvent. A quant. measure can be obtained by scanning the strips with the aid of a suitable densitometer, or colorimetrically after elution of the dye with acetic acid - methanol. E. KAWERAU

1837. **Direct and continuous spectrophotometric assay of β-glucosidase.** B. H. J. Hofstee (Med. Res. Foundation, Palo Alto, Calif., U.S.A.). *Arch. Biochem. Biophys.*, 1955, **59** (2), 398–404.—The method described, which can be used for kinetic studies, is based on the liberation of salicylic acid from salicylic acid β-glucoside (substrate) by β-glucosidase. The amount of salicylic acid formed is determined from the increase in u.v. absorption at 310 mμ. The method is sensitive to 1 μmole of substrate hydrolysed and requires the enzyme extracted from <1 mg of almond for a determination. The influence of pH and substrate concn. is studied and it is shown that glucose, cellobiose and salicin interfere in the method. W. H. C. SHAW

1838. **The use of a selective acetylcholinesterase inhibitor in the estimation of pseudocholinesterase activity in rat brain.** B. J. Bayliss and A. Todrick (Med. School, Birmingham, England). *Biochem. J.*, 1956, **62** (1), 62–67.—The determination of pseudocholinesterase by the use of the selective substrates butyrylcholine or benzoylcholine gives high results when an excess of acetylcholinesterase is present (as in rat brain). Under these conditions, a selective inhibitor for acetylcholinesterase, "62C47" or 1:5-di-(*p*-dimethylaminophenyl)pentan-3-one dimethiodide, is used in the determination of pseudocholinesterase. Similarly the determination of acetylcholinesterase in the presence of pseudocholinesterase is improved by adding ethopropazine methosulphate as a selective inhibitor of the pseudo enzyme. J. N. ASHLEY

1839. **The steroid sulphatase of *Patella vulgata*.** A. B. Roy (Dept. Biochem., Univ. Edinburgh, Scotland). *Biochem. J.*, 1956, **62** (1), 41–50.—Steroid sulphatase is of little general use in the hydrolysis of urinary steroid sulphates before their assay because of its marked specificity—it hydrolyses only the 3β sulphates of 5α- and Δ^5-steroids. The use of enzymic methods in the assay of urinary steroids is criticised. J. N. Ashley

See also Abstracts 1607, 1636, 1715, 1765, 1772, 1786, 1855, 1859, 1862, 1927, 1940.

Drugs

1840. **Use of quantitative oscillographic analysis in pharmacy.** R. Kalvoda (Polarographic Inst. Czech. Acad. Sci., Prague, Czechoslovakia). *Českosl. Farmac.*, 1955, **4** (10), 501–504.—Two methods are described. In one the depths of the oscillographic dips are a direct measure of the concn. of the depolariser for certain concn. regions (about 3×10^{-3} to 1×10^{-5} *M*). A horizontal trace on the oscillographic curve, movable in the vertical plane, is used to read off directly the height of the dip, the concn. then being read off a calibration curve. In the second method the oscillograph of the substance being analysed is adjusted by addition of the material (by titration) till the curve becomes identical with that obtained from a standard solution. Best results are obtained in the concn. region of 3×10^{-4} to 8×10^{-5} *M*, and it is important to synchronise the droplet formation of both dropping electrodes. The methods can be used for the determination of Ni in Co, Pb and Cd in Zn, Pb in brass, etc. The determination of elementary S is described; the oscillograph cathode dip occurs at $-0{\cdot}45$ V and the anode dip at $-0{\cdot}35$ V in glacial acetic acid saturated with Na acetate. In equal parts of chloroform and methanol with 15 per cent. of LiCl, two cathode ($-0{\cdot}46$ V and $-0{\cdot}81$ V) and two anode ($-0{\cdot}55$ V and $-0{\cdot}65$ V) dips occur. Sulphur was satisfactorily determined in methanol, ethanol, chloroform and benzene, and qualitatively detected in thiophen, thiourea, cystine, etc., and various medicinal preparations. A. O. Jakubovic

1841. **Determination of water in drugs by the Karl Fischer method.** G. J. Mulder and J. A. C. van Pinxteren (Pharm. Lab. State Univ. Utrecht, Holland). *Pharm. Weekbl.*, 1956, **91** (2), 33–44. For coloured soln. and most galenicals the visual end-point method of matching the colour of 0·02 *N* iodine soln. is unsuitable and the electrometric dead-stop end-point method is recommended; the method frequently gives greater accuracy than oven-drying or the Pregl tube, especially for the hydrochlorides of the opium alkaloids, for which comparative numerical results are given in tabular form. For soft extracts, 0·5 g of material is triturated with 0·5 g of sand, taken up with 20 ml of absolute methanol, transferred to a 25-ml calibrated flask and is thoroughly mixed by agitation during 30 min. The determination is then made on an aliquot of the clear supernatant liquid. P. Haas

1842. **Oxidised cellulose in ion exchange [of alkaloids]. A preliminary note.** F. M. Freeman (E. R. Squibb & Sons Ltd., Speke, Liverpool, England). *J. Pharm. Pharmacol.*, 1956, **8** (1), 42–45.—Oxidised cellulose has been employed as a cation exchanger in the determination of atropine, strychnine, quinine and codeine. Compared with Amberlite IRC-50 or Decalso, uptake of alkaloid is not greatly affected by the presence of other salts, and recovery is rapid and quant. compared with Amberlite IR-120H. *Procedure*—Prepare a column (5 cm × 1·2 cm) from a slurry of oxidised cellulose ($\simeq$2 g) in H_2O, and wash well with 0·1 *N* H_2SO_4 and H_2O. Transfer the sample (5 ml, containing 5 to 20 mg of alkaloid) to the column and maintain a flow-rate of 1·5 to 2 ml per min. by positive pressure. Wash with H_2O (10 × 5 ml). Elute the alkaloid with 0·1 *N* H_2SO_4 (50 ml) and estimate spectrophotometrically or colorimetrically. A. R. Rogers

1843. **Conductimetric titration of certain alkaloids.** V. V. Udovenko and L. A. Vvedenskaya. *Ukr. Khim. Zh.*, 1954, **20** (6), 684–689.—By conductimetric titration with naphthalene-2-sulphonic acid in acetone solution, the alkaloids present in the following mixtures were separately determined: anabasine - lupinine, anabasine - nicotine, anabasine - nicotine - lupinine, also anabasine - lupinine in the presence of aphylline and aphyllidine. The method depends on the effect of acetone in increasing the difference between the ionisation constants of the bases. It can be applied to the determination of alkaloids in technical products. F. W. Kirkbride

1844. **The photometric determination of alkaloids with the aid of short-wave ultra-violet radiation.** H. Thies, C. H. Sorgenfrey and F. W. Reuther (Inst. Pharm. und Lebensmittchem., Univ., Munich). *Naturwissenschaften*, 1955, **42** (22), 605.—The absorption is measured at 253·7 mμ which is obtained from a low-pressure mercury lamp in conjunction with luminescent substance S17 (*cf.* Wallenfels *et al.*, *Angew. Chem.*, 1953, **65**, 459). At pH 1·9 the molar extinction coefficient, ϵ, for papaverine is $5{\cdot}767 \times 10^4$, for morphine $9{\cdot}486 \times 10^3$ and for ephedrine $1{\cdot}353 \times 10^2$. The Beer - Lambert law is obeyed for extinction values up to E = 1, and measurements are largely independent of temperature. E. Kawerau

1845. **Spectrophotometric determination of heroin [diamorphine] and quinine.** M. J. Pro, W. P. Butler and A. P. Mathers (Alcohol and Tobacco Tax Div. Lab., Intern. Rev. Service, Washington, D.C., U.S.A.). *J. Ass. Off. Agric. Chem.*, 1955, **38** (4), 849–857.—At 297·5 mμ, equimolar concentrations of quinine and hydrolysed diamorphine have almost the same absorption, whilst at 330 mμ the absorption is due entirely to quinine. A 100-mg sample of the mixed alkaloids with 10 ml of anhydrous methanol is filtered through asbestos, the filtrate and methanol washings (40 ml) being diluted to 100 ml with 0·1 *N* NaOH, and of this solution 10 ml are again diluted to 100 ml with NaOH - methanol solution. The extinction is read at 297·5 and 330 mμ. The standard deviation for both constituents is $\pm$ 1·5 p.p.m. on mixtures containing 6 to 30 p.p.m. and 30 to 90 p.p.m. of diamorphine and quinine hydrochloride, respectively. A. A. Eldridge

1846. **Determination of micro quantities of alkaloids by amperometric method. I. Titration of strychnine in an aqueous solution with tungstosilicic acid.** Tadahiko Ogawa (Yokohama Univ., Minamiku, Japan). *J. Chem. Soc. Japan, Pure Chem. Sect.*, 1955, **76** (7), 739–744.—The height of the polarographic reduction wave of tungstosilicic acid (**I**) at $-0{\cdot}70$ V (*vs.* the S.C.E.) in 0·4 *M* KNO_3

soln. is linearly reduced by the addition of strychnine (**II**); **II** does not give a reduction wave at $> -1{\cdot}20$ V. An amperometric titration for 0·04 to 40 mg of **II** is effected with an error of <1 per cent. by the use of standard **I** soln. The use of a simple apparatus, consisting of a calomel electrode and a small cell divided with a sintered-glass filter, was suggested; the time taken for an estimation was decreased to 45 min. K. SAITO

1847. **The fluorimetric determination of ergot alkaloids.** I. Gyenes and K. Szász (Pharm. Factory of Kőbánya, Budapest). *Magyar Kém. Foly.*, 1955, **61** (12), 393–398.—To dried ergometrine maleate (50 mg), protected from light, were added water (10 ml) and 1 per cent. ethanolic ethanesulphonic acid (**I**) (5 ml), diluting finally to 50 ml with water. Aliquots of 3 to 25 ml were diluted to 50 ml with water, transferred to a beaker (diameter 37·5 to 38 mm) and compared at 25° ± 0·3° C with a Zeiss - Pulfrich D fluorescence standard. The values obtained from this dilution series were plotted against the ergometrine base (**II**) content determined by titration. The fluorescence intensity of **II** is 4·5 times that of ergotoxine base, showing that as well as lysergic acid, the substituents also influence the intensity. With increasing molar proportion of the substituents, the fluorescence intensity decreases. Ergotoxine and ergotamine were also examined. A. G. PETO

1848. **The quantitative determination of the salts of ergot alkaloids in anhydrous medium. The determination of the base content and acid component of ergotamine tartrate, ergotamine acid phthalate and ergometrine maleate.** I. Gyenes and K. Szász (Pharm. Factory of Kőbánya, Budapest). *Magyar Kém. Foly.*, 1955, **61** (11), 356–359.—Ergotamine tartrate (**I**) (93 to 116 mg) was dissolved, protected from light, in anhydrous anhydride-free acetic acid (20 ml), and 1 drop of a 1 per cent. soln. of crystal violet in pure acetic acid was added. The soln. was titrated with 0·05 *N* $HClO_4$ in acetic acid to a blue colour (first colour-change). 1 ml of 0·05 *N* $HClO_4 \equiv 29{\cdot}08$ mg of ergotamine base. To determine the acidic component, **I** (194 to 261 mg) was dissolved, protected from light, in pyridine (20 ml) [previously neutralised to a blue colour with K methoxide, in the presence of 0·1 per cent. methanolic thymol blue (0·2 ml)], and the soln. was titrated with 0·1 *N* K methoxide. 1 ml of 0·1 *N* K methoxide $\equiv 7{\cdot}504$ mg of tartaric acid. The error was ± 0·3 per cent. The base content and the acidic component of ergotamine acid phthalate were determined by the same methods. The probable errors were ±0·5 and ±0·8 per cent., respectively. The base content of ergometrine maleate (69 to 216 mg) was determined similarly; the probable error was ± 0·4 per cent. 1 ml of 0·05 *N* $HClO_4 \equiv 16{\cdot}27$ mg of ergometrine. For determining the acidic component (91 to 211 mg), the method described above was used, but with stirring and under nitrogen. The probable error was ±0·9 per cent. 1 ml of 0·1 *N* K methoxide $\equiv$ 5·803 mg of maleic acid. A. G. PETO

1849. **The analysis of lumi-ergot alkaloids.** M. Pöhm (Fabrik A. v. Waldheim, Vienna). *Mikrochim. Acta*, 1955, (5–6), 1016–1018.—A report is given of a spectrophotometric differentiation of the ergot alkaloids from lumi-lysergic acid derivatives. Possible methods of detection are discussed. C. A. SLATER

1850. **The differentiation of roots of *Rauwolfia serpentina* from roots of other *Rauwolfia* species. II. Chemical methods.** D. Banes and J. Carol (Div. Pharmaceut. Chem., F. and D.A., Dept. Health, Education and Welfare, Washington, D.C., U.S.A.). *J. Ass. Off. Agric. Chem.*, 1955, **38** (4), 866–870.—A sample is warmed with ethanol, shaken with chloroform after cooling, and filtered. An aliquot of the filtrate is extracted with two portions of KH_2PO_4 soln. (5 per cent.) and one of $NaHCO_3$ soln. (1 per cent.), and the washed chloroform soln. is mixed with *iso*octane to which are added four chloroform washings of the aqueous fractions. The alkaloids are then extracted with several portions of 2 per cent. citric acid soln. The combined extracts, mixed with ethanol, are made alkaline with NaOH and heated on a steam bath. The alkaline hydrolysate is then washed with chloroform, acidified with HCl, and extracted with chloroform, the chloroform soln. then being extracted with $NaHCO_3$ soln. This, after acidification, is extracted with chloroform, and the u.v. absorption spectrum, determined from 250 to 370 mμ, is compared with the spectra of 3:4:5-trimethoxybenzoic acid and 3:4:5-trimethoxycinnamic acid, the acids formed by the hydrolysis of rescinnamine and reserpine, respectively. A. A. ELDRIDGE

1851. **The quantitative paper-chromatographic separation of digitalis glycosides.** J. Tuzson and G. Vastagh (Staatlich. Hyg. Inst., Budapest). *Pharm. Acta Helv.*, 1955, **30** (12), 444–451.—The exact conditions for making quantitative the paper-chromatographic separation of the digitalis glycosides have been fixed. Low recoveries ($\simeq$90 per cent.) are obtained, particularly in the usual method of assay, but the percentage loss is practically the same for all the glycosides. The lanatoside-C component always contains a hitherto unknown glycoside as impurity. A. R. ROGERS

1852. **The importance of recently discovered glycosides in the chemical assay of digitalis.** F. H. L. van Os and D. H. E. Tattje (Rijksuniv. Groningen, Holland). *Pharm. Weekbl.*, 1955, **90** (27), 901–906.—Recent discoveries of the glycosidic contents of *Digitalis purpurea* are reviewed, and the compositions of 12 such glycosides are given. In connection with determinations described by Tattje and van Os (*Brit. Abstr. C*, 1953, 316) strospeside and digipurpurin are quant. the most important. The presence of strospeside (the cardiotonic activity of which is unknown) will increase the colour with Baljet's reagent by the formation of its decomposition product, gitoxin, but will not affect the Lindewald value. The presence of digipurpurin (which has no cardiotonic activity) will not affect the Baljet value, but its decomposition products, digitoxin and gitoxin, will be found in determinations based on the digitoxose reaction. The probable effects of the presence of these two glycosides on correlations between cardiotonic effects and chemical composition are considered. P. S. ARUP

1853. **Determination of anthracene derivatives in some purgative drugs (buckthorn, cascara, rhubarb, senna).** R. Paris and M.-F. David-Cuny (Lab. de Matière Médicale, Fac. de Pharmacie, Paris). *Ann. Pharm. Franç.*, 1955, **13** (7–8), 488–494.—Free anthracene derivatives in buckthorn are determined by passing the ground bark through a No. 26 sieve, determining the water content and then soaking 0·1 g for 5 min. in 20 ml of water

containing 8 drops of *N* KOH. The suspension is brought to pH 3 (with HCl) and extracted with ether. The ether extract is then extracted with *N* KOH and the extinction is measured at 500 mμ (Bornträger). For combined and total anthracene derivatives, 0·1 g is allowed to stand for 5 min. with 160 ml of water and 3 ml of *N* KOH, and after adding 80 ml of 20 *N* H_2SO_4 is heated over boiling water for 20 min. After extracting with ether and then this extract with *N* KOH, an aliquot is heated for 4 min. over boiling water (for air oxidation of anthranols) and measured colorimetrically as before. To determine combined anthraquinones it is preferable to use a milder acid hydrolysis (10 *N* H_2SO_4) and measure the absorption of the alkaline solution before and after heating in air. The technique for cascara is similar, but yellow pigments must be removed by washing the ether solution with 1 per cent. HCl, and hydrolysis is effected by one treatment with *N* H_2SO_4. For rhubarb the technique is the same as for buckthorn, with slight differences in the concentrations for hydrolysis. The colorimetric determination is standardised with rhein instead of emodin. For senna (leaves) an infusion is used, and yellow pigments have to be removed. The colorimetric measurement is made at 520 to 530 mμ and standardised on a mixture of equal parts of sennosides A and B. Total anthracene derivatives are obtained by hydrolysis with 20 *N* H_2SO_4. E. J. H. Birch

1854. **Estimation of menaphthone by the use of liquid zinc amalgam.** Chozo Yoshimura (Kinki Univ., Fuse, Osaka, Japan). *J. Chem. Soc. Japan, Pure Chem. Sect.*, 1955, **76** (8), 881–883.—The use of liquid zinc amalgam in place of zinc dust or titanous salt for the reduction of menaphthone (**I**) is suggested. When **I** (≃10 mg) is reduced with Zn - Hg in 2 *N* H_2SO_4, the reduction is completed within 5 min. and the product is titrated with ceric sulphate either potentiometrically or with ferroin as indicator. The bisulphite of **I** produces SO_2 on the reduction, which must be removed by suction before the titration. When ammonium molybdate is added to the reduced soln., molybdenum blue, equivalent in amount to the **I**, is produced even in the presence of SO_2; the extinction at 535 mμ is proportional to the amount of **I** within the range 0·01 to 0·2 mg per 10 ml. K. Saito

1855. **The assay of heparin preparations.** J. E. Jorpes (Karolinska Inst., Stockholm). *Acta Pharm. Tox., Kbh.*, 1955, **11** (4), 367–376.—Experience with different methods for the assay of heparin is summarised. Methods requiring thromboplastin or thrombin preparations and those requiring recalcified oxalated or citrated plasma or other artificial coagulation systems are considered unsatisfactory. The U.S.P. XIV and B.P. 1953 methods are criticised, and it is concluded that procedures in which fresh whole blood is used are the most satisfactory for the assay of heparin for clinical use. Details are given of a method requiring fresh ox-blood and of an *in vivo* test depending on the increase in coagulation time of sheep's blood.
W. H. C. Shaw

1856. **A chemical method for vitamin-B_{12} concentration in liver injection.** P. J. Vanmelle (Armour Lab., Kankakee, Ill., U.S.A.). *J. Amer. Pharm. Ass., Sci. Ed.*, 1956, **45** (1), 26–30.—Vitamin B_{12} (**I**) may be determined in liver injection by purification by chromatography on a cation-exchange resin, followed by spectrophotometric assay. Results are in excellent agreement with the U.S.P. microbiological method with *Lactobacillus leichmannii*, but are obtained more rapidly and with greater precision. *Procedure*—Add 10 per cent. KCN (2 ml) to the sample (5 to 50 ml containing 120 to 250μg of **I**) and adjust the pH to 7·5 with citric acid. After ≮3 hr. adjust the pH to 4·0 with citric acid and filter if necessary. Pass at a rate of 2 ml per min. through a 10-cm column of Amberlite XE-97, prepared by previous washes with *N* NaOH, water and citrate buffer of pH 4·0. Elute three bands of coloured impurities with 0·1 *N* HCl, 85 per cent. aq. acetone and 0·1 *N* HCl, respectively. Elute the **I** with 0·1 *N* HCl in 60 per cent. aq. dioxan, and collect 10 ml of the eluate. Pipette 4-ml aliquots into each of two flasks, one containing 10 per cent. aq. KCN (1 ml) and the other alkaline buffer (1 ml) of the same pH. Mix the contents of each flask, and after 30 min. measure the difference $(A_1 - A_2)$ between the extinctions of the two solutions at 578 mμ in 1-cm cells. Then μg of B_{12} per ml of sample =

$$\frac{(A_1 - A_2) \times 1{\cdot}25 \times 10}{0{\cdot}0060 \times \text{ml of sample.}}$$

A. R. Rogers

1857. **Colorimetric determination of erythromycin by means of methyl sulphate.** M. Pesez (Roussel-Uclaf, Paris-Romainville). *Ann. Pharm. Franç.*, 1955, **13** (7–8), 513–516.—Erythromycin is determined by the colour developed in ethyl methyl ketone solution with methyl sulphate. The absorption is similar to that given with erythromycin in 27 *N* H_2SO_4 and has a max. at 480 mμ. Beer's law is obeyed between 50 and 300 μg of erythromycin or its ethylcarbonate. *Procedure*—An amount of a preparation containing ≃100 mg of erythromycin is agitated with 25 ml of ethyl methyl ketone for 15 min. After filtering, 10 ml of the solution are diluted to 100 ml and 0·5 ml of this is made up to 2 ml with ethyl methyl ketone. Methyl sulphate (8 ml) is added and the absorption at 480 mμ is read after 1 hr. at ordinary temp. A comparison is given of the results of determinations by this and a biological method. E. J. H. Birch

1858. **Colorimetric determination of cycloserine, a new antibiotic.** L. R. Jones (Commercial Solvents Corp., Terre Haute, Ind., U.S.A.). *Anal. Chem.*, 1956, **28** (1), 39–41.—Cycloserine reacts with sodium nitritopentacyanoferroate in slightly acid medium to give an intense blue colour which is measured spectrophotometrically. The accuracy is within ±2·0 per cent. and the precision within ±1·0 per cent. in the concn. range 5 to 200 μg. The method is specific for the cycloserine molecule.
G. P. Cook

1859. **Toxicological investigation of micro quantities of barbiturates.** O. A Guagnini, E. E. Vonesch, A. M. Fassi and S. V. Guagnini (Univ. Nacional de La Plata, Buenos Aires). *Mikrochim. Acta*, 1955, (5–6), 1061–1068.—A study of the detection of micro amounts of barbiturates in biological materials has been carried out. The procedure suggested is based on extraction with methanol, followed by ascending paper-chromatography, and identification with the aid of the Parri and Koppanyi reaction. The interferences and special conditions when applying the test to viscera, blood and urine are described. C. A. Slater

1860. **Reactions for the chemical determination of Hostacain, a new local anaesthetic.** A. Häussler and L. Ther (Farbwerke Hoechst A.-G., Frankfurt,

Germany). *Dtsch. ApothZtg*, 1955, **95** (37), 886–888.—Hostacain (the hydrochloride of *n*-butyl-aminoacet-6-chloro-2-methylanilide) (**I**) forms a picrate (m.p. 157° to 158° C) and a ppt. with $K_4Fe(CN)_6$; these tests are recommended for distinguishing the drug from procaine and lignocaine, respectively. Two quant. colorimetric micro-methods are described. (*a*) An aq. dilution (5 ml) containing 25 to 200 μg of **I** is adjusted with 5 ml of acetate buffer to pH 4·6 and treated with 3 ml of 0·1 per cent. bromocresol purple. The mixture is extracted with four 5-ml portions of anhyd. chloroform. The combined extracts are treated with 1 ml of alkaline reagent (1 ml of 15 per cent. NaOH in 99 ml of methanol) and made up to 25 ml. The colour is stable and can be evaluated. (*b*) The same technique is used for determinations with 0·1 per cent. Tropaeolin OO, except that the colour is developed with an acid reagent (1 ml of conc. H_2SO_4 in 99 ml of methanol). The latter method gives higher extinction values. S.C.I. Abstr.

1861. **The ion-exchange chromatographic determination of antihistamines.** S. M. Blaug and L. C. Zopf (Pharmacy Coll., State Univ., Iowa City, U.S.A.). *J. Amer. Pharm. Ass., Sci. Ed.*, 1956, **45** (1), 9–12.—An anion-exchange method is described for the determination of any of 11 antihistamine salts, with recoveries of 98·54 to 101·16 per cent. The method has been extended to the assay of antihistamine tablets, elixirs and ointments. Ephedrine can be separated from tripelennamine by the consecutive use of cation- and anion-exchange resins. *Procedure*—Apply a soln. of the sample (75 mg) in 60 per cent. ethanol (20 ml) to a column of Amberlite IRA-410 (10 ml) previously converted into the hydroxyl form. Elute with 60 per cent. ethanol (50 ml) at a rate of one drop per second and titrate the eluate with 0·1 *N* HCl, with bromocresol green as indicator. Perform a blank determination.
A. R. Rogers

1862. **Paper ionophoresis of some derivatives of 4-hydroxycoumarin.** I. M. Hais (Výzkumný ústav pro farm. a biochem., Prague, Czechoslovakia). *Chem. Listy*, 1955, **49** (5), 709–716.—The following compounds were successfully separated by paper ionophoresis—4-hydroxycoumarin, 3-ethyl-4-hydroxycoumarin, 4-hydroxy-3-phenylcoumarin, 3:3′-methylenebis-4-hydroxycoumarin ("dicoumarol"), di(4-hydroxy-3-coumarinyl)acetic acid, its ethyl ester (ethyl biscoumacetate), glycol monoester and *n*-amyl ester. The spots were detected by their fluorescence in u.v. light or by spraying with diazotised *p*-nitroaniline. From the variation in electrical mobility under the influence of varying pH, the dissociation constants of each substance could be estimated. The ionophoretic behaviour of the 7-hydroxy derivative of ethyl biscoumacetate, isolated from pig's urine, showed that above pH 9 the compound is dissociated in three degrees, in accordance with the suggested structure containing three acidic dissociable groups. The method is useful for the estimation of the p*K* value of the components of complex mixtures (urine extracts) without previous isolation. G. Glaser

1863. **Isolation and detection of 3:3-diethyltetrahydro-2:4-dioxopyridine ("Persedon") for forensic purposes.** F. Dybing (Oslo Univ., Norway). *Acta Pharm. Tox., Kbh.*, 1955, **11** (4), 393–397.—The u.v. absorption, pptn. and colour reactions, and distribution coefficients between immiscible solvents of 3:3-diethyltetrahydro-2:4-dioxopyridine (**I**) are investigated. Small amounts of **I** can be isolated by descending one-dimensional chromatography on Whatman No. 1 paper, with toluene - acetic acid - water (10:4:5), benzene - acetic acid - water (10:4:5) and *n*-butanol - acetic acid - water (4:1:5) as solvents, or on formamide-impregnated paper developed with $CHCl_3$ containing 1 per cent. of ethanol. After development and drying, the paper is sprayed with 3 per cent. aq. NaOH and examined moist in u.v. light; 0·1 μg of **I** gives a blue - green fluorescence. R_F values are 0·62, 0·71, 0·93 and 0·75 with the respective solvents. Identification is by elution with 0·1 *N* H_2SO_4 and u.v. absorption. The behaviour of **I** in the Stas - Otto process is discussed. W. H. C. Shaw

1864. **Gravimetric determination of 2-benzyl-4:5-dihydroiminazoline [tolazoline].** J. Blažek and Z. Stejskal (State Inst. for the Control of Drugs, Prague, Czechoslovakia). *Českosl. Farmac.*, 1955, **4** (10), 506–508.—Two methods, in which Reinecke's salt (**I**) and tungstosilicic acid (**II**) are used, are described. The first ppt. as weighed contains tolazoline (**III**) and **I** in the molecular proportion of 1 to 1, so that 1 g of it corresponds to 0·4099 g of **III** hydrochloride. The second ppt. is in the ratio of 4 mol. of **III** to 1 mol. of **II**, 1 g containing 0·2232 g of **III** hydrochloride. The pptn. with **I** is carried out at room temp. and is accurate to within 0·5 per cent., that with **II** at 70° C with an error of <2·5 per cent. Both ppt. are dried at 100° C.
A. O. Jakubovic

1865. **The "melting-point" of Cyclospasmol and the quantitative determination of small amounts of 3:3:5-trimethylcyclohexyl phenylglyoxylate therein.** R. F. Rekker, M. J. E. Ernsting and W. T. Nauta (N.V. Koninklijke Pharm. Fabr. v/h Brocades-Stheeman & Pharm., Amsterdam, Netherlands). *Pharm. Weekbl.*, 1956, **91** (1), 1–6.—The varying m.p. of Cyclospasmol (3:3:5-trimethyl-*cyclo*hexyl mandelate) (**I**) is partly due to contamination with 3:3:5-trimethyl*cyclo*hexyl phenylglyoxylate (**II**) but mainly due to isomers produced by using inactive mandelic acid and low-melting inactive 3:3:5-trimethyl*cyclo*hexanol as starting materials. By repeated fractional crystallisation from ethanol, an ester of m.p. 84° C is produced. Three methods are described for the quant. determination of **II** in **I**. In the spectrophotometric method, the extinctions in ethanol are determined at 252, 258 and 264 mμ in a 10-mm cell against ethanol as a blank. The polarographic determinations are made with a manual polarograph, drop time 2·82 sec. The analysis soln. is 500 mg of **I** in 80 ml of ethanol made up to 100 ml with aq. 0·5 *N* KCl soln. The diffusion current is measured at a potential of ≃1·50 V. A rapid routine method described involves precipitation with 2:4-dinitrophenylhydrazine (**III**). To a soln. of 50 mg of **I** in 1 ml of aldehyde-free and ketone-free methanol, 1 drop of a soln. of 120 mg of **III** in 5 ml of 30 per cent. $HClO_4$ is added. After being mixed, the soln. is left to stand for 1 hr., with occasional scratching of the vessel. The absence of a ppt. indicates a percentage of **II** <0·3 per cent. O. M. Whitton

1866. **Colorimetric determination of disulfiram.** B. Salvesen and L. Domange (Lab. Contrôle des Médicaments, Fac. Pharmacie, Paris). *Ann. Pharm. Franç.*, 1955, **13** (7–8), 499–502.—In the presence of copper turnings in many solvents tetramethyl- and tetraethyl-thiuram disulphide (disulfiram) develop a yellow colour with a max. at

434 mμ, characteristic of the >N·CS·S·S·CS·N< group. *Procedure*—To a 10-ml sample of a solution in acetone is added 0·45 g of copper turnings (treated with HNO_3 and washed with H_2O, alcohol and ether), and the mixture is left at room temp. for 7 hr. Two or three measurements of extinction are made towards the end of the reaction and the max. reading obtained (which lasts for ≃ 0·5 hr.) is recorded. Calibration data are given for acetone and also for methanol solutions. E. J. H. BIRCH

1867. **Estimation of camphor in pharmaceutical preparations.** H. C. Mital and K. N. Gaind (Pharmaceutics Dept., Med. Coll., Amritsar, India). *J. Pharm. Pharmacol.*, 1956, **8** (1), 37–41.—A volumetric method, in which hydroxylamine hydrochloride (**I**) is used, is given for the estimation of camphor in pharmaceutical preparations. Recoveries range from 96 to 102 per cent. *Procedure*—For spirit, liniment and soln. of camphor in turpentine oil, reflux the sample (containing 0·2 g of camphor) with 95 per cent. aldehyde-free ethanol (20 ml), 4 per cent. **I** in 90 per cent. aldehyde-free ethanol (10 ml) and $NaHCO_3$ (0·3 g) for 4 hr. and cool. Rinse the condenser with light petroleum (20 ml), titrate with 0·2 *N* HCl, with dimethyl yellow as indicator, and then with 0·2 *N* KOH, with phenolphthalein as indicator. Carry out a control estimation omitting the camphor, and calculate from the difference between the titres of KOH. For liniment of soap, liniment of turpentine and ammoniated liniment of camphor, dilute the sample (containing 2·5 g of camphor) to 50 ml with steam-distilled turpentine oil, add H_2O (50 ml), acidify with dil. H_2SO_4 to methyl orange and steam-distil. Separate the oily layer of the distillate, wash the condenser and separator with ethanol (50 ml each) and extract the aq. layer with solvent ether (30 ml). Bulk the oil, alcoholic soln. and ether, dilute to 250 ml with ethanol and use a 20-ml aliquot for the estimation. A. R. ROGERS

1868. **The identification of narcotics and anaesthetics in toxicological analysis.** J. Bäumler (Kantonalem Lab., Basle, Switzerland). *Mitt. Lebensmitt. Hyg., Bern*, 1955, **46** (5), 431–441.—The melting points, general properties and reactions are given for 29 new narcotics and anaesthetics including 5-ethyl-5-*cyclo*heptenyl barbituric acid (Medomin), 3:3-diethyl-2:4-dioxo-5-methylpiperidine. (Noludar), and amylobarbitone (Amytal). Some suggestions are given for the isolation of these substances during toxicological analysis. C. A. SLATER

1869. **Paper-chromatographic detection of 1-naphthylthiourea (ANTU) for forensic purposes.** F. Dybing (Oslo Univ., Norway). *Acta Pharm. Tox., Kbh.*, 1955, **11** (4), 388–392.—Isolation and identification of 1-naphthylthiourea (**I**) in organic matter is by extraction of the material with 95 per cent. ethanol, filtration, distillation of the solvent *in vacuo*, pptn. with absolute ethanol, solution in methanol - water (1:1) and extraction of fat with *n*-heptane. After distillation of methanol, **I** is extracted with chloroform and the extract is purified by descending one-dimensional chromatography on formamide-impregnated filter-paper developed with chloroform (R_F value, 0·44). After drying, the paper is sprayed with 3 per cent. aq. NaOH and examined under u.v. light. A blue fluorescent spot is detectable with 2 μg of **I**. Further identification is by a colour reaction and by u.v. absorption after elution of **I** with ethanol. W. H. C. SHAW

See also Abstracts 1862, 1891, 1913.

Food

1870. **A rapid method for the estimation of calcium and magnesium in foods and other phosphate-containing substances.** A. K. Gosh and K. L. Roy (Univ. Coll. Sci. Technol., Calcutta, India). *Naturwissenschaften*, 1955, **42** (24), 644–645.—A modified stannic chloride procedure is used to remove phosphate before titration with EDTA (disodium salt). Food material is ashed and an aliquot of an acidic solution of the ash containing 10 to 15 mg of Ca is neutralised with aq. NH_3 and then made slightly acid with a drop of conc. HCl. Three ml of 30 per cent. stannic chloride solution are added followed by 10 ml of 5 per cent. hydroxylamine solution. The volume is made up to 50 ml with water, the solution is warmed to hydrolyse the tin salt, then boiled for a few minutes. After filtering through Whatman No. 41 paper the filtrate is titrated with EDTA (disodium salt) for calcium and magnesium in the usual way. If iron and aluminium contents are high they must be removed as hydroxides after the phosphate separation. E. KAWERAU

1871. **Application of the anthrone reagent to the estimation of starch in cereals.** K. M. Clegg (Rowett Res. Inst., Aberdeen, Scotland). *J. Sci. Food Agric.*, 1956, **7** (1), 40–44.—The method has been applied to the analysis of the starch and sugar content of pure starches, cereals, cereal by-products and experimental poultry rations. Finely ground material (0·2 g) is weighed into a 50-ml centrifuge tube, mixed with water (5 ml) and hot 80 per cent. alcohol (25 ml) and centrifuged; the alcohol solution is decanted, and the extraction process is repeated. The two alcohol extracts are combined and the alcohol is evaporated under reduced pressure. The remaining cloudy aq. fraction is diluted with water to make a final concn. of sugar ≡ ≃ 100 μg of glucose per ml. To extract the starch the residue is treated with 5 ml of water and 6·5 ml of 52 per cent. $HClO_4$ and stirred, 20 ml of water are added and the mixture is centrifuged; the supernatant liquor is poured into a 100-ml flask. Water (5 ml) is added to the residue and the $HClO_4$ extraction is repeated. The contents of the tube are washed into the 100-ml flask, made up to vol. with water and filtered. An aliquot portion is diluted to give a concn. of sugar ≡ 100 μg of glucose per ml. The anthrone reagent (10 ml) is added to each of the following test samples: (1) the blank (2 ml of water), (2) test extract alone (containing 1 ml of diluted extract and 1 ml of water, and (3) test extract plus glucose standard [containing 1 ml of diluted extract and 1 ml (100 μg) of glucose standard]. The tubes (closed with rubber stoppers containing a capillary tube) are heated in a bath of boiling water for 12 min. then cooled to room temp. The intensity of the colour is read with an absorptiometer with an Ilford 607 filter. The anthrone - sugar complex has max. absorption at 630 mμ. S.C.I. ABSTR.

1872. **Analysis of barley proteins.** E. Waldschmidt-Leitz and H. Brutscheck (Tech. Hochsch., Munich). *Brauwissenschaft*, 1955, **8** (12), 278–282.—A complete analysis of barley proteins was carried out by the latest methods, including chromatography and electrophoresis. The amino acids and their amounts in the four main barley proteins are reported, as well as in other proteins, including the various cereal prolamines, and the chill haze of beer. It was concluded that the insoluble chill haze of beer consisted mainly of a

peptide which is formed by the hydrolysis of the hordein. Comments are made on the relation of protein composition to barley type.

G. H. BAKER

1873. **Determination of potassium, sodium, calcium and magnesium [in molasses] by combined paper chromatography and polarography.** M. Vender (Výzkumný ústav cukrovar., Prague, Czechoslovakia). *Chem. Listy*, 1955, **49** (5), 771–772.—Evaporate the sample (2 g) with conc. H_2SO_4, dissolve the residue, after igniting it at 600° C, in H_2O (5 ml), exchange SO_4'' for Cl′ by passing the soln. through an anion-exchange column, wash the column with H_2O and reduce the combined eluates to 5 ml. Chromatograph 0·05 ml of the soln. on Whatman No. 1 paper, the spots being detected by spraying with 1 per cent. $AgNO_3$, followed by exposure to sunlight. Cut the paper into pieces, extract each zone with 0·1 *N* H_2SO_4 and determine the content of Cl′ by polarography. From the results, calculate the content of the separated cations. A correction for impurities in the filter-paper must be applied. G. GLASER

1874. **The nitrogen content of meat and the calculation of the meat content of meat products.** J. F. Reith, M. J. N. Hofsteede and W. Langbroek (Univ. Utrecht, Holland). *J. Sci. Food Agric.*, 1955, **6** (6), 317–323.—The authors query the correctness of the Stubbs and More (SM) formula as modified by the Soc. of Publ. Analysts and other Analytical Chemists (*Analyst*, 1940, **65**, 257), based on the assumption that the mean nitrogen content of "lean" beef is 3·4 per cent., that of "lean" pork 3·6 per cent., and that of "mixed meat" 3·5 per cent. Ten publications giving both the nitrogen and the fat contents of fresh beef and pork samples are quoted, to illustrate the widely divergent SM figures they yield. Original analyses of samples of Dutch pork (both fresh and salted), giving average SM figures as low as 3·29 (winter samples of salted pork) and 3·40 (summer samples of fresh pork), are reported. A. TESSLER

1875. **Some physical properties of milk. I. Density.** W. D. Rutz, C. H. Witnah and G. D. Baetz (Kansas State Coll., Manhattan, U.S.A.). *J. Dairy Sci.*, 1955, **38** (12), 1312–1318.—From a study of the effects of temp. and homogenisation on the observed density of milk it is concluded that, between 18° and 44° C, density is a linear function of temp. to within 0·0005 g per ml. The temp. coefficient in this range is 0·00038. Homogenisation pressures up to 300 lb per sq. in. caused a highly significant increase in density with each increase in pressure, but pressures between 500 and 3000 lb per sq. in. had no significant effect.

W. H. C. SHAW

1876. **A detergent test for the milk-fat content of dairy products. I. Milk, cream and ice-cream.** O. S. Sager, G. P. Sanders, G. H. Norman and M. B. Middleton (Dairy Products Section, Eastern Utilisation Res. Branch, Agric. Res. Service, U.S. Dept. Agric., Washington, D.C., U.S.A.). *J. Ass. Off. Agric. Chem.*, 1955, **38** (4), 931–940.—A modification of the BDI detergent test for butter fat (Sager and Sanders, *Milk Ind. Found. Conv. Proc.*, Lab. Sect., 1952, **4**, 29) is described. A sharper meniscus results from the addition of 2 per cent. of $NaHCO_3$ to the detergent - tetraphosphate reagent; the samples are shaken after 2 min. in the bath, then shaken twice during a further 15-min. immersion in boiling water, and a tempering period of 5 min. is sufficient. Milk and ice-cream gave results in agreement with those obtained by the Babcock and Röse - Gottlieb tests. With cream the curved bottom meniscus may be flattened by treating the bottles with silicone; results are then equal to those afforded by the Babcock test and 0·25 per cent. higher than those given by the Röse - Gottlieb test. A. A. ELDRIDGE

1877. **Measurement of oxidation of dried milk products with thiobarbituric acid.** C. G. Sidwell, H. Salwin and J. H. Mitchell, jun. (Quartermaster Food and Container Inst. Armed Forces, Chicago, U.S.A.). *J. Amer. Oil Chem. Soc.*, 1955, **32** (1), 13–16.—Steam-distillation of an acidified slurry of milk powder and the application of the thiobarbituric acid test for oxidation products (see *Anal. Abstr.*, 1956, **3**, 1887) has advantages over previously published methods. Milk powder (15 g), water (75 ml) and 3 *N* HCl (7 ml) are steam-distilled until 50 ml of distillate are collected. The test described earlier is applied to 20 ml of the distillate, correcting the reading by a reagent blank. Results are expressed as extinctions. The results agree with estimates of oxidation from flavour. N. E.

1878. **Pectic products in fruit. Determination of galacturonic acid in tomatoes and its changes on storage.** B. Borenstein, E. F. Stier and C. O. Ball (Agric. Expt. Sta., Rutgers Univ., New Brunswick, N.J., U.S.A.). *J. Agric. Food Chem.*, 1955, **3** (12), 1041–1044.—Tomatoes (a 1-kg sample) were cut into thin slices and dropped into 2750 ml of boiling 95 per cent. ethanol to give 70 per cent. ethanol; the mixture was boiled, cooled and filtered. 1:3-Dihydroxynaphthalene reagent (2 ml) was added to a sample of soln. containing galacturonic acid (2 ml) and 1·83 ml of conc. HCl in a 25-ml flask. The mixture was heated on a water bath at 91° C for 45 min., then cooled for 10 min., and the violet colour extracted by shaking with 2 ml of 95 per cent. alcohol and 17 ml of ether. The extinction of the ether extract was determined at 575 mμ with a water-cooled Beckman DU spectrophotometer. The method permitted the analysis of galacturonic acid soln. containing $<0·0550$ mg per ml to be carried out with a precision within 0·0004 mg per ml. S.C.I. ABSTR.

1879. **Examination of bromo and iodo compounds as preservatives in beverages.** R. G. Eeckhaut. *Fermentatio*, 1955, (2), 71–85.—The use of org. compounds of Br and I, in particular bromoacetates and iodoacetates, as preservatives in beverages, and methods of detection, are reviewed. A modification of the author's earlier procedure for determining org. Br in beverages is described. The sample (200 ml) is acidified with 10 ml of 25 per cent. v/v H_2SO_4 and extracted with ether (3 × 30 ml). The ether extracts are combined and mixed with 10 to 20 ml of 94 to 96 per cent. alcohol and, after separation, the ether layer is added to $\simeq$1 g of MgO and 10 ml of water and evaporated to dryness before being heated at 450° C for 30 min. Water (7 to 8 ml) is added to the cooled product which, after standing for 30 min., is filtered. To the filtrate (10 ml) are added 8 drops of HCl (sp. gr. 1·19), 8 drops of aq. K_2CrO_4 (10 per cent.) and 2 ml of H_2SO_4 (50 per cent., by vol.). After 5 min., 2 ml of fuchsine reagent [10 ml of 0·1 per cent. aq. soln. mixed with 100 ml of dil. H_2SO_4 (1:20) and set aside for 1 hr.] and 2 ml of alcohol-free $CHCl_3$ are

added. The presence of Br is indicated by a violet coloration of the $CHCl_3$ layer; a photocolorimetric measurement can be made at 560 mμ. A similar procedure is described for the detection of org. compounds of I. To the 10-ml filtrate (see above) are added 1 ml of starch soln. (0·5 per cent.), 1 ml of glacial acetic acid and 0·2 ml of aq. $NaNO_2$ (2 per cent.); the development of a blue coloration in about 30 min. indicates the presence of I. (43 references.) S.C.I. ABSTR.

1880. **Determination of fermentable sugars in beer by paper chromatography.** H. R. Held, R. H. Garratt and W. D. McFarlane (Canadian Breweries Ltd., Toronto, Canada). *Proc. Amer. Soc. Brew. Chem.*, 1955, 167–173.—The chromatographic procedure previously described for the quant. estimation of carbohydrates and of total fermentable sugars in wort (McFarlane and Held, *Ibid.*, 1953, 67; *cf.* also *Anal. Abstr.*, 1955, **2**, 2230) has been modified so that it can be used with beer. The beer sugars are collected on a small area of the paper so that direct colorimetric analysis can be made. The fermentable-sugar content of a series of beers varied from 0·24 to 0·86 g per 100 ml, expressed as glucose. S.C.I. ABSTR.

1881. **The qualitative testing of gelatin for use in the fining of drinks.** H. Rentschler and F. Hauser (Eidg. Versuchsanstalt, Wädenswil, Switzerland). *Mitt. Lebensmitt. Hyg., Bern*, 1955, **46** (5), 405–414. —The ability of various gelatins to precipitate tannins from wines and ciders has been examined. It is shown that there is no relation between the glutin content of a gelatin and its ability to precipitate tannin. C. A. SLATER

1882. **Dulcin and its detection in beverages.** R. G. Eeckhaut. *Fermentatio*, 1955, (2), 86–93.—The chemical and pharmacological properties of dulcin and the various procedures that have been described for its determination are reviewed. In the method recommended, 50 ml of sample are clarified with 5 ml of Pb acetate (20 per cent., or 30 to 40 per cent. if necessary), 2 ml of aq. NH_3 (sp. gr. 0·91) are added, the mixture is set aside for 30 min. and filtered. The filtrate is mixed for 1 min. with ethyl acetate (20 ml) and allowed to stand for 15 min. The layers are separated and the upper, ester, layer is then shaken for 5 min. with 5 g of anhydrous Na_2SO_4 to dehydrate thoroughly. A 10-ml aliquot of the ester extract is mixed with 1 ml of HNO_3 (sp. gr. 1·40); a yellow colour is produced which reaches max. intensity after 2 hr. The mixture is allowed to stand for at least 18 hr. and the extinction is measured (the max. absorption is at 410 mμ). The amount of dulcin is read from a graph prepared with solutions of 1 to 6 mg of dulcin per litre. (23 references.) S.C.I. ABSTR.

1883. **Separation and identification of *cyclo*hexylsulphamate, dulcin and saccharin by paper chromatography.** L. C. Mitchell (F. and D.A., Dept. Health, Education and Welfare, Washington, D.C., U.S.A.). *J. Ass. Off. Agric. Chem.*, 1955, **38** (4), 943–946.—Paper is spotted with 0·1 *M* solutions of the sweetening agents and developed with a mobile solvent consisting of 10 ml of ethyl acetate and 10 ml of conc. aq. NH_3 made up to 100 ml with acetone. After drying, the chromatogram is sprayed with 0·005 *M* $AgNO_3$ in ammoniacal ethanol, dried in the air, and then sprayed with 0·0005 *M* pyrogallol in ethanol. The white areas of the silver salts of *cyclo*hexylsulphamate and saccharin are outlined with pencil, and then the chromatogram is sprayed with conc. HNO_3 and dried overnight whereby the dulcin spot becomes visible.

A. A. ELDRIDGE

1884. **Standardisation of analytical methods for evaluation of activated carbons used in wine and oil industries.** A. Zucca. *Ann. Fac. Agr. Univ. Cattol. S. Cuore*, 1955, **52**, Ser. 2, 225–233.—Suggested methods for the evaluation of activated carbons for use in the wine and oil industries include those for impurities, pH, apparent density, effect of carbon on colour, acetic, tartaric and volatile acids, ash, alcohol and alkalinity of wines; oleic acid content, peroxide value and Kreis reaction of oils; and ignition loss, ash, pH, matter sol. in water and acid, Fe, Cl′, SO_4'', Zn, PO_4''' Ca, Mg and SiO_2 of the carbon. F. R. PAULSEN

1885. **Calorimetric studies on the melting behaviour of fats and oils.** L. Riedel (Bundesforschungsanstalt Lebensmittelfrischhaltung, Karlsruhe, Germany). *Fette u. Seifen*, 1955, **57** (10), 771–781. —A specially constructed adiabatic calorimeter is used to determine the temperature coefficient of the specific heat of 27 fats over the range −55° C to +55° C. It is suggested that the method may have applications in fat analysis. E. HAYES

1886. **Determination of analytical characteristics in oxidised oils.** K. Meier, R. Milewsky and H. Wentzel (Lab. Anstrichforsch. Tech. Univ. Berlin-Charlottenberg). *Fette u. Seifen*, 1955, **57** (9), 702–710.—The effect of oxidation products on the analytical characteristics of methyl 9:12-linoleate, methyl 10:12-linoleate and linseed-oil fatty alcohols, oxidised by shaking with oxygen at 30° C, is studied. In the presence of peroxides the carbonyl numbers obtained are too high, but the true values can be found by the use of a correction based on the known peroxide content; epoxides and other oxygenated compounds do not interfere. When the hydroxyl number is determined by acetylating with acetic anhydride in pyridine and measuring the unconsumed acetic anhydride, peroxides do not interfere but epoxides do. Ester numbers are too high because of the presence of peroxides. The total iodine value (Woburn) gives the true number of double bonds; the hydrogenation number, determined with dioxan as solvent, agrees with the theoretical value when allowance is made for peroxides present. Alkoxyl oxygen can be determined by subtracting oxygen of other groups from the total oxygen found by elementary analysis. Acid values are not affected by the oxygenation products. E. HAYES

1887. **The use of thiobarbituric acid as a measure of fat oxidation.** C. G. Sidwell, H. Salwin, M. Benca and J. H. Mitchell, jun. (Quartermaster Food and Container Inst. Armed Forces, Chicago). *J. Amer. Oil Chem. Soc.*, 1954, **31**, 603–606.—Dissolve a 3·0-g sample of fat in 10 ml of C_6H_6 or CCl_4 in a Maisel-Gerson tube, add 10 ml of thiobarbituric acid (**I**) reagent, shake in a horizontal position for 4 min. at about 125 oscillations per min., and withdraw the aq. layer into a test-tube (25 mm × 200 mm). Immerse the tube in boiling water for 30 min., cool, transfer a portion of the contents to a cuvette and read the extinction at 530 mμ against water. The **I** reagent is prepared by adding 0·67 g of **I** to ≃75 ml of glacial acetic acid in a 100-ml beaker, heating on a steam bath for several min. and adding 2 ml of conc. HCl to facilitate solution.

The soln. is cooled and made up to 100 ml with glacial acetic acid. CHEM. ABSTR.

1888. **Impurity measurement in fats. Spectrophotometric determination of melamine and formaldehyde in lard.** R. C. Hirt, W. R. Doughman, R. G. Schmitt and S. T. Moore (Amer. Cyanamid Co., Stamford, Conn., U.S.A.). *J. Agric. Food Chem.*, 1955, **3** (12), 1044–1046.—For the determination of melamine, lard (15 g) dissolved in *iso*octane (100 ml) was extracted with dil. HCl (10 ml of 0·1 *N* HCl). After shaking in a separating funnel, the bottom (acid) layer was removed, and the extraction was repeated with another 10-ml portion of acid. The extracts were combined and extinction measurements were made at 235 and 260 mμ. The concentration of melamine and/or resin (each has approx. the same absorptivity at 235 mμ) was calculated from a given equation. For the determination of formaldehyde, a sample of lard (15 g) was dissolved in 30 ml of *iso*octane and extracted with water (4 × 3 ml). The first and second extracts were combined as were the third and fourth and the two sets were examined separately. The water extract (of ≯6 ml) was treated with 1 ml of 1 per cent. phenylhydrazone hydrochloride and allowed to stand for 10 min. Then 0·5 ml of 5 per cent. $K_3Fe(CN)_6$ and 2 ml of 12 *N* HCl were added in rapid succession. The soln. was made up to 10 ml and the colour was measured after 10 min. at 520 mμ. The concentration of formaldehyde was calculated from a given equation. S.C.I. ABSTR.

1889. **Determination of residual light petroleum in extracted oils and extraction meals.** A. Purr and A. Hettich (Inst. Lebensmitteltech. u. Verpackung, Munich, Germany). *Fette u. Seifen*, 1955, **57** (10), 782–789.—Methods of determining residual light petroleum in extracted oils and extraction meals are reviewed, and a new method is proposed. For oils, a sample mixed with glacial acetic acid is heated on a sand-bath under reduced pressure until all the acetic acid has been distilled. An aliquot of the distillate is mixed with water and the petroleum is removed by a stream of air; the air stream is passed through a "Perlick" apparatus in which catalytic combustion of the petroleum at alternate junctions of a thermopile produces a current which is related to the petroleum content of the air. Extraction meals are boiled for two hours with a mixture of acetic acid and water (11:39), a stream of air being passed through the mixture; the petroleum in the issuing air is entrained in propionic acid, cooled in ice - salt mixture. The petroleum in an aliquot of this solution is then determined as described. The proposed method gives results agreeing closely with those obtained by the simpler method of Pardun (*Seifensiederztg*, 1947, **73**, 101, 121, 141). The light petroleum can also be determined gravimetrically after adsorption on carbon. E. HAYES

1890. **Tables for the rapid application of the Morton - Stubbs correction in vitamin-A assay.** A. R. Rogers (Anal. Dept., Allen and Hanburys Ltd., Ware, Herts., England). *Analyst*, 1955, **80**, 903–906.—The equation for the Morton - Stubbs correction for irrelevant absorption in the spectrophotometric assay of vitamin A is re-arranged and combined with the generally accepted factor, 1900, for converting $E^{1\%}_{1\,cm}$ to units of vitamin A per g. The equation then takes the form of the sum of two products, and tables are given for the values of these products. Calculation from the experimental data is thus reduced to the addition of two figures obtained from the tables and division by the concn. and the cell width. The tables are applicable in the B.P. methods for halibut-liver oil and concentrates in *cyclo*hexane and the unsaponifiable fraction of cod-liver oil in *cyclo*hexane, and in the U.S.P. method for the unsaponifiable fraction in *iso*propyl alcohol. A. O. JONES

1891. **Comparison of results of determining ascorbic acid in industrial preparations by the iodate and indophenol methods.** V. A. Devyatnin, V. V. Zvorÿkina and N. M. Stol'nikova. *Trudy Vsesoyuz. Nauch. Issledovatel. Vitamin. Inst.*, 1954, **5**, 196–200; *Referativnŷĭ Zh., Khim.*, 1955, Abstr. No. 40,494.—The error in the determination of ascorbic acid by the dichlorophenolindophenol method is 0·57 to 0·64 per cent. and that by the iodate method is 0·04 to 0·19 per cent. The latter method is recommended for determinations of ascorbic acid in plums and in tablets as well as in cryst. products. G. S. SMITH

1892. **Chemical estimation of vitamin-E activity in cereal products. II. Millers' offals and compound animal foods.** P. W. R. Eggitt and L. D. Ward (Univ. Birmingham, England). *J. Sci. Food Agric.*, 1955, **6** (6), 329–337.—Samples of millers' offals and compound feeding-stuffs were analysed and their tocopherol pattern was determined, introducing improvements developed since the basic technique was published in Part I (*J. Sci. Food Agric.*, 1953, **4**, 569). The biological value of the various members of the tocopherol group is discussed. A. TESSLER

See also Abstracts 1769, 1791, 1856, 1917.

Sanitation

1893. **Determination of injurious gases in air, by Reich's method.** E. Kraus (Centr. Inst. Foundry Technol., Leipzig, Germany). *Chem. Tech., Berlin*, 1955, **7** (9), 552–554.—According to Reich's determination of SO_2 in air, the gaseous mixture is passed from a measuring cylinder through a known iodine - starch solution in an absorption flask until decolorisation occurs. The content of SO_2 is then calculated from the remaining vol. of gas. A modified method, based on this principle, is described and applied for the determination of H_2S, SO_2, Cl_2, NH_3, HCl and O_3. S.C.I. ABSTR.

1894. **Determination of carbon monoxide in air.** D. P. Senderikhina. *Inform.-metod. Materialŷ Gos. Nauch. Issledovatel. Sanit. Inst.*, 1954, (5), 21–24; *Referativnŷĭ Zh., Khim.*, 1955, Abstr. No. 40,401.—Air is passed through absorption tubes containing successively silica gel, pumice moistened with conc. H_2SO_4, and an absorbent for CO_2. The carbon monoxide is then burnt to form CO_2, and the CO_2 is absorbed in 0·005 *N* $Ba(OH)_2$. The excess of $Ba(OH)_2$ is titrated with 0·005 *N* HCl. With 500 ml of air followed by 100 ml of purified air the error of the determination is ±1·6 per cent. The method is sensitive to 0·0014 mg of CO. G. S. SMITH

1895. **Determination of arsine in air.** M. V. Alekseeva. *Inform.-metod. Materialŷ Gos. Nauch. Issledovatel. Sanit. Inst.*, 1954, (5), 16–18; *Referativnŷĭ Zh., Khim.*, 1955, Abstr. No. 40,375.—The air, at a rate of 0·5 litre per min., is passed for 30

min. through two absorbing vessels containing 5·5 to 16·5 ml of a mixture (5:5:1 by vol.) of solutions of 0·32 g of $KMnO_4$ per litre, 0·3 ml of conc. H_2SO_4 per litre and bromine water. The combined solutions are then treated with 1 per cent. H_2O_2 solution to destroy the permanganate and evaporated to dryness. The residue is dissolved in 25 ml of 25 per cent. H_2SO_4 solution and the solution is transferred to a flask. The ordinary Gutzeit procedure is then followed, comparing the colour produced by mercuric bromide with standards.

G. S. SMITH

1896. **Use of synthetic resin ion-exchange materials for the determination of atmospheric fluorides.** J. P. Nielsen and A. D. Dangerfield. *Arch. Ind. Hlth*, 1955, **11** (1), 61–65.—A new method for determining fluoride compounds in the atmosphere for contents of 1 to 10 parts per 10^9 is based on the separation of F' by an ion-exchange resin. A known vol. of air is passed through a wash-bottle containing water or dil. HCl and the soln. is percolated through a column of Duolite-A41 resin, which retains the F'. The ions are recovered by washing the resin first with 0·1 *N* then with 0·01 *N* NaOH. The alkaline soln. is neutralised to alizarin red with *N* HCl. The F' are titrated in the presence of a buffer (chloroacetic acid) with thorium nitrate, a red coloration denoting the end-point.

LIGHT METALS BULL. ABSTR.

1897. **Determination of hydrazine and ammonia in air.** H. McKennis, jun., and L. B. Witkin (Dept. Pharmacol., Med. Coll. Virginia, U.S.A.). *Arch. Ind. Hlth*, 1955, **12** (5), 511–514.—A quant. method suitable for routine analysis of air samples containing both hydrazine and ammonia is described. The contaminated air is bubbled through 20 ml of 0·1 *N* H_2SO_4. To a suitable aliquot of this solution (containing $\simeq 50$ μg of NH_3) are added 10 ml of satd. K_2CO_3 and the NH_3 is removed by aeration into 20 ml of 0·1 *N* H_2SO_4. The NH_3 is determined with Nessler reagent. Another aliquot (equivalent to $\simeq 1$ μg of hydrazine) is diluted to 5 ml and 0·5 ml of *p*-dimethylaminobenzaldehyde soln. (0·2 g in 5 ml of 2 *N* H_2SO_4) is added. After 5 min. the colour is compared with that of standard solutions. The hydrazine may also be determined in the alkaline solution from which NH_3 has been removed.

N. M. WALLER

1898. **Determination of pyridine in air and in fibres.** B. A. Gaukhman. *Inform.-metod. Materialy Gos. Nauch. Issledovatel. Sanit. Inst.*, 1954, (5), 28–30; *Referativnyĭ Zh., Khim.*, 1955, Abstr. No. 40,475.—To determine pyridine in air, the air at a rate of 1 litre per min. is passed through an absorber containing 4 ml of 0·01 *N* NaOH. The solution after transference to a colorimeter tube is mixed with 1 ml of 0·1 *N* NaOH, 50 mg of $(NH_4)_2SO_4$, 0·15 ml of BrCN (prepared by decolorising 5 to 10 ml of saturated bromine water with 10 per cent. KCN solution added dropwise) and 0·2 ml of a saturated solution of aniline in water. The liquid is shaken and the colour is compared after 10 to 12 min. with that of standards prepared from a solution of pyridine containing 0·01 mg per ml. To determine pyridine in fibres, 10 g of the material are extracted with 50 ml of 0·1 *N* NaOH for 10 min. The colour given by the reagent with the filtered extract is compared with that of standards prepared by adding known amounts of pyridine to extracts of similar material not containing pyridine. The method is sensitive to 0·0005 mg of pyridine in 5 ml of solution. The error is $\pm$0·001 mg.

G. S. SMITH

1899. **The determination of potassium in water and effluents as potassium tetraphenylboron.** E. Zymny. *Prakt. Chem.*, 1955, **6** (12), 327–329.—The sample (1 litre) is concentrated to 100 ml and acidified with acetic acid; 10 ml of 3 per cent. aq. $NaB(C_6H_5)_4$ are added and after 5 min. the pptd. $KB(C_6H_5)_4$ is filtered off, dried at 120° C, and weighed. Crystallisation of the ppt. is improved by adding a few drops of aq. (10 per cent.) $AlCl_3$; a similar addition improves the stability of the reagent, which is $\not>$ 14 days. Potassium in the ppt. may be determined volumetrically by dissolving it in acetone (10 ml) and 2 *N* acetic acid (5 ml), adding 0·1 *N* KBr (1 ml) and aq. eosin, and titrating with 0·1 *N* $AgNO_3$. The KBr improves the end-point and must be allowed for in calculating the result. There is no interference by Li, Ca, Ba and Sr, but Rb, Cs and NH_4^+ are pptd. When K and NH_4^+ are both present, the mixed ppt. is weighed, dissolved in acetone, and heated on the water bath with 20 ml of 2·5 per cent. aq. NaOH until the acetone and NH_3 are expelled; after cooling, the residual $KB(C_6H_5)_4$ is again filtered off, washed, dried at 105° C, and weighed. The method is quick and accurate; the reagent is recoverable (*cf. Brit. Abstr. C*, 1953, 337).

A. R. PEARSON

1900. **A new method for the colorimetric determination of oxygen dissolved in water.** Motoharu Tanaka (Univ. Nagoya, Japan). *Mikrochim. Acta*, 1955, (5–6), 1048–1056.—The method described is based on the restoration of the colour to a reduced solution of manganese formaldoxime.

C. A. SLATER

1901. **Polarographic determination of B.O.D.** B. L. Rosenthal and R. L. Donlan. *Sanitalk*, 1955, **3** (2), 7–12.—The apparatus and experimental procedure for a new polarographic method for the determination of the B.O.D. and the concentration of dissolved O of sewage and waste waters are described. The operating potential and the calibration procedure are given and the factors affecting the validity of the calibration curve are indicated. The method requires less time than the Winkler method; small B.O.D. bottles are used.

WATER POLLUTION ABSTR.

1902. **The biochemical oxygen demand test: a note on variable results from the use of stored standard dilution water.** A. B. Wheatland and R. G. Smith (Water Pollution Res. Lab., Stevenage, Herts., England). *Analyst*, 1955, **80**, 899–900.—Experiments have shown that the common practice of keeping a stock of dilution water may give rise to B.O.D. values very different from those obtained when freshly prepared dilution water is used. Analysis of the stored dilution water showed that practically all the NH_3 originally added had been oxidised to NO_3', indicating the probable presence of nitrifying bacteria which encouraged nitrification in the diluted sewage during the five-day test. Further expt. showed that standard dilution water may be stored for a short period in vessels cleaned with H_2CrO_4 and thoroughly rinsed, but the practice of "topping up" the stock should be avoided.

A. O. JONES

1903. **Measurement of tritium in water samples.** J. F. Cameron (Isotope Div., Atomic Energy Res. Estab., Harwell, England). *Nature*, 1955, **176**,

1264.—Tritium-bearing water vapour at saturation vapour pressure is introduced as part of the filling of an internal gas counter. The filling is completed with 10 to 15 cm pressure of a standard mixture (9:1) of argon and alcohol. The counters used are type GA.10.M., by Twentieth Century Electronics, in conjunction with an external quench unit, type 1014A. Plateaux are obtained >400 V and a slope <2 per cent. per 100 V. The threshold voltage is about 1400 V. The counters are not sensitive to light and are readily cleaned by pumping and flushing with pure water vapour. G. A. BASSETT

1904. **Interference by industrial wastes in the Mohr test for chlorides.** M. J. Taras (Dept. of Water Supply, Detroit, Mich., U.S.A.). *Wat. & Sewage Wks*, 1955, **102** (11), 442–446.—The extent of interference with the Mohr test by sulphur compounds from oil-refinery wastes was studied and interfering concentrations of thiols, sulphides, thiourea and thioglycollic acid are tabulated. The interference can be removed in some cases by u.v. irradiation and in others by evaporation. The former method, provided that adequate irradiation is effected, removes practically all the sources of interference, whilst evaporation removes volatile or thermally unstable thiols and sulphides.
S.C.I. ABSTR.

1905. **Determination of aniline in water.** A. I. Kuper. *Inform.-metod. Materialy Gos. Nauch. Issledovatel. Sanit. Inst.*, 1954, (5), 31–32; *Referativnyĭ Zh., Khim.*, 1955, Abstr. No. 40,500.—The sample of water (5 ml) in a colorimeter tube is mixed with 1 ml of 4 per cent. aq. chloramine-T solution, 1 ml of 3 per cent. aq. phenol solution and 0·5 ml of 2 per cent. NaOH solution. After 15 to 20 min. the colour is compared with that of standards. The minimum amount of aniline that can be determined is 0·1 mg per litre. G. S. SMITH

Agriculture and Plant Biochemistry

1906. **Rapid volumetric method for determining cellulose in studies with rumen micro-organisms *in vitro*.** T. V. Hershberger, O. G. Bentley and A. L. Moxon (Ohio Agric. Exp. Sta., Wooster, U.S.A.). *J. Anim. Sci.*, 1955, **14**, 725–730.—Residual cellulose from the digestion test is transferred to a 10-ml graduated centrifuge tube and washed with water. To the residue are added acetic acid (6·5 ml) and HNO_3 (1·5 ml) and, after stirring the suspension, the tube is placed in a boiling-water bath for 20 min., stirring being repeated. The tube is subsequently cooled and centrifuged for 5 min. at 1000 r.p.m. The top of the cellulose plug is gently levelled with a glass rod and the tube is recentrifuged for 5 min. and the vol. of cellulose is measured. The method has the same order of accuracy (standard deviation ± 1·58) as the Crampton - Maynard gravimetric procedure. A. G. POLLARD

1907. **Soil contamination of herbage samples.** A. Thompson and A. M. Raven (Sch. Agric., King's Coll., Univ. Durham, England). *J. Sci. Food Agric.*, 1955, **6** (12), 768–777.—Sources of error in mineral analyses of herbage, especially for trace elements, from the inclusion of small amounts of soil are discussed. Methods of removal of contaminating material by washing, and indirect methods of assessing contamination, *e.g.*, by the determination of Al, are reviewed. In a proposed procedure for the determination of Al, the plant material (1 g) is ashed, then heated with Na_2CO_3 (0·5 g). Conc. HNO_3 (1 ml) and water (3 ml) are added to the cooled product, and the soln. is evaporated to dryness overnight. Water (10 ml) and HNO_3 (1:50) (10 ml) are added and the mixture is heated on a water bath for 20 min. Iron, indicated by a reddish-brown coloration of the silica, is dissolved by heating with thioglycollic acid (0·5 per cent.) (10 ml). The soln. is filtered, the ppt. being washed alternately with hot water and HNO_3 (1:50) (2 × 10 ml), and finally with cold water, and the filtrate is made up to 100 ml. An aliquot portion (containing ≃10 to 15 μg of Al) is transferred to a 10-ml graduated test-tube and 1 ml each of HNO_3 (1:50) and thioglycollic acid (0·5 per cent.) and 2 ml of ammonium aurintricarboxylate reagent (0·75 g of ammonium aurintricarboxylate, 200 g of ammonium acetate, 15 g of gum acacia and 189 ml of conc. HCl, dissolved separately, then combined and made up to 1500 ml) are added. The soln. is made up to 10 ml, heated in a water bath at 90° C for 15 min., then transferred to a water bath at 20° C. The extinction of the red aluminium lake is measured spectrophotometrically at 535 mμ, between 6 and 48 hr. after colour development. A calibration curve is prepared similarly.
S.C.I. ABSTR.

1908. **A manometric method for the determination of carbonate in soils.** A. J. Cavell and S. Graham (Min. Agric., Fish & Food, Nat. Agric. Advis. Serv., Wolverhampton, England). *Lab. Practice*, 1955, **4** (12), 485–487.—The apparatus illustrated is easy to operate and is suitable for routine use. The completion of the reaction between the acid and carbonate is shown visually by the level of Hg in the manometer remaining stationary. With a 10-g sample the working range is 0 to 6 per cent. of $CaCO_3$ in the soil; other ranges can be obtained by adjustment of the sample wt. O. M. WHITTON

1909. **The use of ion-exchange resins in the determination of potassium [in fertilisers].** W. M. Ashton and J. H. Williams (Univ. Coll., Aberystwyth, Wales). *J. Sci. Food Agric.*, 1955, **6** (6), 311–316.—A column of the chloride form of the resin Amberlite IR-4B was used for the removal of sulphates and phosphates from salts and compound fertilisers, and the potassium was then determined by the perchlorate method. The results compared favourably with figures obtained by more orthodox methods, *e.g.*, removal of sulphates as $BaSO_4$, preliminary pptn. of K as cobaltinitrite, or even flame photometry. A. TESSLER

1910. **Determination of certain elements in agricultural limestones by group separation and spectrography.** P. Chichilo, A. W. Specht and C. W. Whittaker (Agric. Res. Service, U.S. Dept. Agric., Beltsville, Md., U.S.A.). *J. Ass. Off. Agric. Chem.*, 1955, **38** (4), 903–912.—Aluminium, Co, Cu, Fe, Mn, Mo, V and Zn in limestone are separated from most of the Ca and Mg by pptn. with oxine and tannic acid from solutions buffered at pH 5·9 with ammonium acetate. The ashed ppt. are dissolved in HCl and portions of the solution are examined by arc spectrography with addition of indium chloride. Transmittancies for Al 2652·5, Co 3453·5, Cu 3274·0, Fe 3286·8, Mn 2939·3, Mo 3132·6, V 3184·0, Zn 3345·0 and In 2932·6 A are measured. For Al, Fe and Zn the comparison standard technique is used, whilst for the other elements In is used as an internal standard.
A. A. ELDRIDGE

1911. **Determination of water-soluble phosphorus in fertilisers by repeated washing and by digestion.** W. M. Hoffman, B. M. Olive and W. L. Hill (Soil & Water Conservation Res. Branch, U.S. Dept. Agric., Beltsville, Md., U.S.A.). *J. Ass. Off. Agric. Chem.*, 1955, **38** (4), 888–897.—Factors influencing the results of phosphorus determinations by washing the samples repeatedly with separate portions of water and by digesting them once with a specified volume of water were investigated. The digestion procedure is the more precise, but gives results for commercial fertilisers which are lower by up to 0·45 per cent. of P_2O_5 than those afforded by washing. The differences are, however, no greater than those likely to result from allowable variations in specifications of the official (A.O.A.C.) procedure. The effects of variations in the conditions of filtration are tabulated. A. A. ELDRIDGE

1912. **Photometric determination of total phosphorus in feeding-stuffs and fertilisers.** G. F. Rickey and A. W. Avens (N.Y. State Agric. Exp. Sta., Cornell Univ., Geneva, N.Y., U.S.A.). *J. Ass. Off. Agric. Chem.*, 1955, **38** (4), 898–903.—To prepare the required molybdovanadate reagent, a cooled solution of 40 g of ammonium molybdate in 400 ml of water is added to a cooled solution of 1 g of ammonium vanadate in 300 ml of water to which 200 ml of conc. HNO_3 have been added. The mixture is diluted to 1 litre. A sample (0·25 to 1 g according to the percentage P guaranteed) is boiled with 20 ml of HNO_3, evaporated to <5 ml, and boiled to fuming with 10 ml of $HClO_4$ (70 to 72 per cent.). After cooling, diluting and filtering, an aliquot is diluted and treated with molybdovanadate reagent. After 15 min. the transmittance is read, with a spectrophotometer or photoelectric colorimeter, and compared with that of standard solutions. The method is rapid and sufficiently accurate for routine analysis.
A. A. ELDRIDGE

1913. **The separation of Aureomycin (chlortetracycline) and oxytetracycline in feeds by paper chromatography.** A. V. Stiffkey and W. L. Williams (American Cyanamid Co., Pearl River, N.Y., U.S.A.). *J. Ass. Off. Agric. Chem.*, 1955, **38** (4), 870–874.—Paper sheets, prepared by dipping in a phosphate buffer solution at pH 3·0 and drying, are spotted with an acid acetone extract of the feed. The sheet is developed, using the descending technique, with *n*-butanol saturated with water, the temperature (between 24° and 28° C) being maintained constant. The sheets are then placed on a medium (peptone 6 g, trypticase 4 g, yeast extract 3 g and beef extract 1·5 g, diluted to 1 litre and 1 per cent. of agar added) inoculated with *Bacillus cereus*. The plates are cooled to 4° C for 1 hr., and then incubated at 20° to 25° C for 24 hr. The method distinguishes between chlortetracycline and oxytetracycline, and the feed can then be assayed quant. Chlortetracycline gives considerably larger zones of inhibition than an equal amount of oxytetracycline. A. A. ELDRIDGE

1914. **Analysis of organic mercurials.** V. H. Chambers, F. R. Cropper and H. Crossley (Murphy Chem. Co. Ltd., Wheathampstead, Herts., England). *J. Sci. Food Agric.*, 1956, **7** (1), 17–28.—A Progress Report on the work of the Joint Committee of the Ministry of Agriculture and the Association of British Insecticide Manufacturers on the determination of total Hg in organo-mercurials is presented. Selected methods were tried on suitable mixtures and a relatively simple method was developed involving reduction with Zn in glacial acetic acid, filtration, dissolution of the zinc amalgam in HNO_3 and titration with thiocyanate. In many instances the zinc amalgam need be washed only with water, but when BHC is present the amalgam must be washed thoroughly with hot glacial acetic acid; when chalk is present, it is advisable to wash the amalgam with dil. acetic acid to control the effervescence. S.C.I. ABSTR.

1915. **Colorimetric determination of chloranil in fungicide preparations.** D. P. Johnson (Analytical Division, N. Carolina Dept. Agric., Raleigh, N.C., U.S.A.). *J. Ass. Off. Agric. Chem.*, 1955, **38** (4) 946–949.—Standards are prepared from a solution of 0·3 g of chloranil in 100 ml of benzene, of which 0·2 to 1·2-ml portions are treated with 10 ml of 2 per cent. NaOH soln. at 25° C, mixed for 15 sec., treated with 1 ml of 40 per cent. acetic acid and diluted to about 45 ml with water. After separation the aq. phase is diluted to 50 ml, the benzene rising above the mark. After shaking and keeping for 10 min. the benzene is decanted and the transmittance of the aqueous solution is measured at 545 mμ. The standard curve so obtained is used in determining the chloranil content of a sample of which a quantity representing about 0·15 g is dissolved in benzene to 100 ml. A. A. ELDRIDGE

1916. **Crop protection products approval scheme. IV. Analysis of dinoseb and MCPA weedkillers.** K. Gardner (Fisons Pest Control, Ltd., Harston, Cambridge, England). *J. Sci. Food Agric.*, 1956, **7** (1), 8–11.—Three methods were considered for the analysis of preparations of dinoseb (2-*sec.*-butyl-4:6-dinitrophenol) (**I**)—(1) titanous chloride titration, depending on quant. reduction of the nitro groups to primary amine in acid or buffered soln., (2) pptn. of the complex formed by **I** with nitron and weighing the dried ppt., and (3) a solubility method in which the crude **I** is separated from the weedkiller formulation by extraction of the acidified soln. with ether, followed by dissolution in four times its wt. of alcohol, and cooling to −20° C, when pure **I** crystallises. An aliquot portion of the filtered supernatant liquor is weighed, then evaporated and the residue weighed. From the wt. of impurities, together with the known solubility of pure **I** in alcohol at −20° C, the purity of the crude **I** can be calculated. Pure **I** is identified by its m.p. or by that of its nitron complex. For MCPA weedkillers (active ingredient, 4-chloro-2-methylphenoxyacetic acid) the main procedures considered were—(*i*) isotope-dilution methods, (*ii*) i.r. methods, (*iii*) u.v. spectrophotometric methods, and (*iv*) chromatographic methods. The chromatographic methods involve the use of kieselguhr - phosphate buffer as static phase, ether - chloroform as mobile phase, a 16-mm column through which the solvent runs at 2·5 ml per min., and titration of the fractions with standard alkali.
S.C.I. ABSTR.

1917. **Pesticide residues, determination of residues of OO-diethyl O-(6-methyl-2-*iso*propyl-4-pyrimidyl) phosphorothioate [Diazinon] in milk.** R. C. Blinn and F. A. Gunther (Univ. Calif., Citrus Expt. Sta., Riverside, Calif., U.S.A.). *J. Agric. Food Chem.*, 1955, **3** (12), 1013–1016.—The use of Diazinon for the control of houseflies in dairy barns resulted in the development of a method to determine residual amounts present in milk. A modification of the

Harris procedure is described. The Diazinon is hydrolysed to 6-methyl-2-*iso*propyl-4-pyrimidinol; interfering substances are then extracted with ether and light petroleum from strongly acidic and basic aq. soln. The pyrimidinol is isolated from a weakly acidic soln. by chloroform extraction and is then determined by its absorption at 272 mμ. To minimise the formation of refractory emulsions the whole milk was freeze-dried; the process is described and illustrated. The residual milk solids were extracted with medium-boiling light petroleum and the Diazinon was extracted with four 35-ml portions of methyl cyanide. Test recoveries were 63 to 70 per cent. S.C.I. Abstr.

See also Abstracts 1766, 1787, 1878.

5.—GENERAL TECHNIQUE AND LABORATORY APPARATUS

General

1918. **An easily prepared analytical glass filter.** L. Erdey and I. Buzás (Polytech. Univ., Budapest). *Magyar Kém. Foly.*, 1955, **61** (12), 443–447.—The prep. and use of a simple, chemically stable and easily prepared glass filter-funnel is described in detail. It consists of a woven glass ribbon, wound round a glass rod. Filters of various porosities can be prepared. The ppt. is dried to const. wt. in the filter in 10 to 20 min., by heating in an aluminium block while drawing air or gas through it. The ppt. can also be heat-treated in a stream of gas. A. G. Peto

1919. **A simple method for ultrafiltration through Cellophane.** H. H. Lausen (Copenhagen Univ., Denmark). *Acta Pharm. Tox., Kbh.*, 1955, **11** (4), 353–354.—The simple glass apparatus described requires no specialised equipment and is readily adaptable for particular requirements. W. H. C. Shaw

1920. **A new semi-micro ebulliometric apparatus for the determination of molecular weight.** B. Bobranski (Inst. of Pharm. Chem. Med. Acad., Wrocław, Poland). *Roczn. Chem.*, 1955, **29**, 461–467.—A new semi-micro ebulliometric apparatus, based on a principle similar to that of Sucharda and Bobranski (*Chemikerztg*, 1927, **51**, 568) is described and illustrated. By using electric heating and separate tubes for liquid and gaseous solvent phases a satisfactory stability of temp. readings is achieved. The average accuracy of determinations is ±5 per cent. S.C.I. Abstr.

1921. **Simple and robust aspirator for flue-gas sampling.** C. H. Buck (Radiation Ltd., Birmingham, England). *J. Sci. Instrum.*, 1955, **32** (12), 489.—The lower opening of a glass aspirator is connected via rubber tubing to a football bladder. The upper opening of the aspirator is connected through a tap to the source of gas. The aspirator is filled with confining liquid, and the bladder is placed below the bottle when the gas is drawn into the bottle. G. Skirrow

1922. **A micro-apparatus for the detection of hydrogen fluoride.** P. Reckendorfer (Bundesanstalt für Pflanzenschutz, Vienna). *Mikrochim. Acta*, 1955, (5–6), 1008–1011.—A modification of the apparatus developed by Feigl and Krumholtz is described. C. A. Slater

1923. **Accurate rapid micro-analysis of gases.** H. Feichtinger. *Arch. Eisenhüttenw.*, 1955, **26** (3), 127–130.—An apparatus for the routine micro-analysis of gas mixtures, especially those obtained in the hot vacuum-extraction of metals, is described and illustrated; it permits the rapid and accurate analysis of 0·1 to 1 cu. cm of gas. The gas (measured by micrometer pump) passes irreversibly and quant. from one reaction chamber to the next, while the reagents always remain in their own compartments, which are isolated by Hg or oil. J. P. Stern

1924. **Report on recommended specifications for microchemical apparatus. Alkoxyl.** Committee on Microchemical Apparatus (Div. Anal. Chem., Amer. Chem. Soc.). *Anal. Chem.*, 1956, **28** (1), 112–115.—Recommended specifications are described for a Clark-type apparatus, which can be used for the determination of alkoxyl groups, either by volumetric or gravimetric means. G. P. Cook

1925. **Simple instrument for titrimetry without visual indicators. Gasometric titrations. X. Cerimetry, chlorimetry, nitrate, nitrite, hydroxide and carbonate determination.** O. R. Gottlieb (Min. Agric., Rio de Janeiro, Brazil). *Anal. Chim. Acta*, 1955, **13** (6), 531–542.—The apparatus is similar to that previously described (*Anal. Abstr.*, 1956, **3**, 286), but the manometer is replaced by a capillary tube that delivers the evolved gases to an inverted water-filled burette of 20 ml capacity. In titrations in which gases are evolved the vol. of gas obtained are plotted against the vol. of titrant added. The graph consists of two straight lines intersecting at the point where gas evolution ceases, *i.e.*, at the end-point of the titration. This method is used for the titration of NaN_3 with 0·1 *N* $Ce^{\cdots\cdot}$, for the titration of $NH_4^{\cdot}$ or hydrazine with $Ca(ClO)_2$ (0·13 *N*) in the presence of Br' and for the titration of sulphamic acid with 0·1 *M* $NaNO_2$. Nitrate can thus be determined by decomposing it with an excess of sulphamic acid and titrating the excess. When $Fe^{\cdot\cdot}$ or $Fe(CN)_6^{''''}$ are titrated with 0·1 *N* $Ce^{\cdots\cdot}$ in the presence of hydrazine sulphate, evolution of N commences at the end-point, which is marked by the intersection of the gas-evolution graph with the titrant axis. Arsenite can be titrated similarly, with hydrazine sulphate or NaN_3 as indicator, or with 0·1 *M* $Ca(ClO)_2$, with hydrazine sulphate as indicator. In the titration of Na_2CO_3 with 0·1 or 0·25 *N* HCl, two end-points are obtained, corresponding to the conversion of CO_3'' into HCO_3' and the complete decomp. of HCO_3', respectively. Azide and hydrazine in the same soln. can be titrated separately with 0·1 *N* $Ce^{\cdots\cdot}$ because the azide reacts almost completely first and the slopes of the gas-evolution curves differ. Gasometric titrations occupy ≃ 10 min. and are comparable in accuracy with standard methods.

XI. Dichromatometry. O. R. Gottlieb. *Ibid.*, 1956, **14** (1), 24–27.—The direct gasometric titration of ions that do not release a gas by reaction with the titrant can be effected by adding a compound that does liberate a gas with excess of titrant. The end-point of the main reaction is then obtained from the gas-titration curve for the gasometric indicator. For example, titrations of $Fe^{\cdot\cdot}$ and $Sn^{\cdot\cdot}$ (and possibly $Ti^{\cdot\cdot\cdot}$) with 0·1 *N* $K_2Cr_2O_7$ in 6 *N* H_2SO_4 can be made by addition of ≃5 ml of 0·1 *M* hydrazine sulphate per 5 ml of sample solution, with ferroin as internal indicator. The precision and accuracy compare well with the

conventional titration, with an external indicator.
W. C. JOHNSON
W. J. BAKER

1926. **Flask stoppers in volumetric analysis.** W. Böhm (Greifenhagener Str. 16, Berlin, Germany). *Z. anal. Chem.*, 1955, **147** (6), 415–417.—A stopper is described that enables atmospheric oxygen to be excluded from the contents of a flask. The stopper consists of a pear-shaped bulb through which runs a glass tube from a carbon dioxide generator. Round the side of the bulb are 6 to 8 grooves which, while permitting the escape of gases, are slanted to prevent the loss of soln.
J. H. WATON

1927. **Qualitative "test-tube" paper chromatography.** G. Gorbach, H. Demmel and S. Gräsböll (Tech. Hochschule, Graz, Austria). *Mikrochim. Acta*, 1955, (5–6), 1037–1047.—A study has been made of the "test-tube" method for the separation and determination of amino acids. R_F values are given for 19 amino acids in various solvent-systems. *n*-Propanol - water (4:1) and ternary mixtures of *tert.*-butyl alcohol - ethyl methyl ketone - water (3:2:1) and *tert.*-butyl alcohol - methanol - water (4:4:1) gave the best separations. C. A. SLATER

1928. **Application of vapour-phase chromatography.** J. G. Keppler (Unilever Res. Lab., Vlaardingen, Holland). *Chem. Weekbl.*, 1955, **51** (51), 911–914.—Various methods of vapour-phase chromatography, with particular reference to detection methods, are discussed. Heat conductivity on platinum wire is found most suitable for analysis of mixtures containing fatty alcohols and fatty acid esters. At column temp. of 160° C, C_8 to C_{14} alcohols are well separated; for C_{16} and C_{18}, 185° C is more suitable. The method is claimed to be quicker than fractionation and equally accurate.
H. A. FISHER

Optical

1929. **Use of carbon electrodes for the spectrographic analysis of solutions.** I. M. Nagibina and I. A. Gel'derman. *Sb. statei Leningr. in-ta tekhnol mekhan. i optiki*, 1954, (11), 108–115; *Referativnyĭ Zh., Khim.*, 1955, Abstr. No. 18,951.—For spectrographic analysis, standard solutions obtained by dilution of primary standard solutions are recommended. As an example the calculation and subsequent preparation of a binary solution of Mn and Mg, where Mg serves as an internal standard at a concn. of 1 g in 100 g of solution, are described. Sparking curves obtained with various methods of introducing the solution into the discharge on carbon electrodes are studied, namely (1) impregnation method, (2) placing a drop of the solution on the carbon electrode previously treated with a 3 per cent. solution of polystyrene in benzene, and (3) allowing drops to fall on to the electrode surface. The second method gives a regular curve for the dependence of the log. of relative intensity on the log. of concn. The first and third methods are less applicable to accurate quant. analysis. Calibration curves of relative intensities for binary solutions of Mn - Mg, Fe - Cu and Cd - Zn over the relative concn. range 0·01 to 300 per cent. are given.
G. S. SMITH

1930. **Flame spectrograms. I. Common metals.** H. Watanabe and K. K. Kendall, jun. (Beckman Instruments, Inc., Fullerton, Calif., U.S.A.). *Applied Spectroscopy*, 1955, **9** (3), 132–140.—The Beckman model DU flame photometer, when fitted with the photomultiplier attachment and the spectral energy recording attachment (*Anal. Abstr.*, 1956, **3**, 1934), permits a complete flame spectrum from 320 to 1100 mμ (or 283 to 540 mμ for Mg) to be recorded in 14 min. from 30 ml of solution. Flame spectrograms of 18 common elements, obtained by this technique, are reproduced for reference purposes. Spectrogram data with possible interferences are listed. P. T. BEALE

1931. **Spectrochemical analysis with the rotating electrode.** J. P. Pagliassoti [Standard Oil Co. (Indiana), Whiting, Indiana, U.S.A.]. *Applied Spectroscopy*, 1955, **9** (4), 153–158.—Applications of the rotating-disc solution method are described. The sample, in solution form, is picked up on a rotating graphite wheel which dips into it and is then excited by a spark or intermittent arc discharge passing between the upper edge of the disc and a graphite counter electrode. In oil solution, 2 p.p.m. of P, 0·2 p.p.m. of V and 0·05 p.p.m. of Ag can be detected, and precision for the determination of additives in new oils is within 2 to 3 per cent. of content. Similar reproducibility is obtained for the analysis of catalysts, steel and other materials in solution. P. T. BEALE

1932. **Spectrochemical analysis of solutions by means of bored electrodes.** L. Erdey, E. Gegus and E. Kocsis (Inst. Gen. Chem., Tech. Univ., Budapest). *Acta Chim. Acad. Sci. Hung.*, 1955, **7** (3–4), 343–360.—The reactions of certain elements which result in gaseous products have been used for the spectrochemical analysis of the elements concerned. The compounds studied were methyl borate, germanium hydride and germanium chloride. These compounds were prepared in a micro-distillation apparatus and were led through a bored electrode directly into the spark- or arc-gap. The method proved to be suitable for germanium. This technique can also be used for the general analysis of solutions. The solution to be investigated is sprayed directly into the arc- and spark-gaps. The spraying apparatus is described in detail and the suitability of the method is demonstrated by examples.
C. A. SLATER

1933. **Spectrographic determination of an element by addition of known amounts of the element.** J. M. Codina Vidal (C.S. de I.C., Barcelona). *An. Real. Soc. Esp. Fis. Quim.*, 1955, **51B** (11), 613–618.—The principles of the method of spectrographic analysis in which a known amount of the element is added to the substance under examination, and the intensity of the relevant spectral lines reduced by means of a rotating sector to the value before the addition, are outlined. Various procedures and their accuracy are compared. The method is useful for the determination of impurities when no sample of the pure substance is available.
L. A. O'NEILL

1934. **Adaptation of Beckman DU spectrophotometer to direct recording.** L. Cahn (Beckman Instruments, Inc., Fullerton, Calif., U.S.A.). *Anal. Chem.*, 1956, **28** (1), 141–142.—A circuit is described for converting the normally supplied null-balance amplifier to a negative feed-back d.c. amplifier capable of operating a conventional mV recorder or micro-ammeter. Spectra may be scanned by operating a recorder in conjunction with a wavelength drive. P. T. BEALE

1935. **A manometric actinometer for the ultra-violet spectrum.** L. P. Zill (Oak Ridge Nat. Lab., Tennessee, U.S.A.). *Arch. Biochem. Biophys.*, 1955, **59** (2), 473–483.—By the method described, a conventional Warburg manometric apparatus fitted with quartz reaction-vessels can be standardised for photochemical and biological irradiation studies requiring u.v. irradiation. The vol. of the mixture of CO and CO_2 produced by the photochemical decomposition of 0·05 *M* oxalic acid with uranyl sulphate (0·01 *M*) by the radiation from a mercury lamp is proportional to the energy involved and can be measured directly in the apparatus. Variations in the ratio of CO and CO_2 with different concn. of oxalate and uranyl ions and with low- and high-pressure mercury lamps are studied.

W. H. C. Shaw

1936. **A heated infra-red cell.** H. C. Mattraw (Knolls Atomic Power Lab., General Electric Co., Schenectady, New York, U.S.A.). *Applied Spectroscopy*, 1955, **9** (4), 177.—An easily constructed cell is described for use with materials which are corrosive or of low vapour pressure, or both, at room temperature. An external heating-coil permits investigations to be made up to 125° C. Windows of AgCl or KRS-5 are recommended.

P. T. Beale

1937. **A low-temperature infra-red cell assembly.** G. J. Janz and W. E. Fitzgerald (Rensselaer Polytech. Inst., Troy, N.Y., U.S.A.). *Applied Spectroscopy*, 1955, **9** (4), 178.—The standard demountable liquid cell is modified to permit the investigation of liquid - solid transformations at low temperatures down to −115° C. An outer jacket is flushed with a controlled stream of the coolant—liquid air or N. An inner jacket carries a stream of dry N to prevent condensation of atmospheric moisture on the windows.

P. T. Beale

1938. **Infra-red microspectroscopy. IV. A double-beam infra-red microspectrometer.** E. R. Blout and M. J. Abbate (Polaroid Corp., Cambridge, Mass., U.S.A.). *J. Opt. Soc. Amer.*, 1955, **45** (12), 1028–1030.—A Perkin - Elmer double-beam spectrometer has been converted to an infra-red microspectrometer by incorporating a reflecting microscope system in the sample beam. Compensation for the increased path length and the inevitable transmission losses is achieved by including a gas cell and a wire-gauze filter in the reference beam. Spectra are illustrated of a single crystal of androsterone and of 1 μg of benzil in CCl_4 soln. Satisfactory resolution is obtained in about one-fifth of the time necessary for single-beam microspectrometry (*Brit. Abstr. C*, 1952, 1263).

B. S. Cooper

1939. **Visual colorimetry in ultra-violet light.** I. A. Stolyarova. *Vestnik Leningrad. Univ.*, 1954, (2), 141–150; *Referativnŷĭ Zh., Khim.*, 1955, (9), Abstr. No. 16,541.—Colorimetry in the u.v. region gives new possibilities in the determination of several elements. A Pulfrich visual u.v. colorimeter is used; filters "UVS-3" or "UVS-1" with a K_2CrO_4 soln. separated the lines 365 mμ or 318 mμ needed for the work from the radiation of mercury lamp "PRK-4." The use of luminescent screens provided visual observations. The following methods were evolved—(1) The determination of Ce in apatites at 365 mμ at concn. from 4×10^{-7} to 4×10^{-6} g per ml (error 0·5 to 3 per cent.). (2) The determination of Fe in cobalt salts, Cu - Ni alloys, and bronze at 318 mμ at concn. from 2×10^{-4} to 152×10^{-4} g per ml. There is no interference from Co, Ni, Cu, Cr, Mn, alkali and alkaline-earth metals, Al, Zn, and Cd; phosphates must be absent. Best results are obtained in sulphate soln.; the error is from 1·2 to 5·3 per cent. (3) The determination of small quantities of V (0·01 to 0·02 per cent.) in the presence of comparatively large amounts of Cr at 318 mμ. The application of the method to the determination of V in ordinary steels is indicated.

C. D. Kopkin

1940. **A simple d.c. fluoriphotometer for detecting uranium.** M. F. Milligan and R. J. Watts (Los Alamos Sci. Lab., New Mexico, U.S.A.). *Nucleonics*, 1955, **13** (6), 83–86.—Small aliquots of urine suspected of containing U are evaporated and fused with NaF, by using the general procedure of Newman *et al.* (*J. Biol. Chem.*, 1948, **173**, 41) and Price *et al.* (*Brit. Abstr. C*, 1953, 303). Details are given of a simple electronic circuit for use with a photocell to measure the intensity of the 555-mμ fluorescence line, excited when the fluoride "button" is placed under ultra-violet light.

E. N. Jenkins

1941. **A new type of spectrometer for determination of the refractivity of liquids and gases.** H. L. Lorentzen (Univ. Kjem. Inst., Blindern-Oslo, Norway). *Acta Chem. Scand.*, 1955, **9** (10), 1724.—Displacement of a slit image, produced by a lens system and a hollow prism filled with the liquid or gas under test, is used as a measure of its refractivity.

P. T. Beale

1942. **Some analytical applications of diffuse reflectance measurements.** R. B. Fischer and F. Vrátný (Indiana Univ., Bloomington, U.S.A.). *Anal. Chim. Acta*, 1955, **13** (6), 588–593.—Measurements made with a Beckman spectrophotometer with a reflectance attachment show that the diffuse reflectance spectrum of malachite green, obtained from a paper dipped in a soln. of the dye and dried, is closely similar to the transmission spectrum of a soln. of the dye. The reflectance and transmission spectra of a copper ammine show less similarity. Reflectance measurements for copper ammine, $Fe(SCN)_3$, malachite green and Congo red are plotted against the concn. of soln. used to moisten the paper. These graphs provide an evaluation of the procedure as an analytical method. Similar measurements are made with mixtures of powdered solids—Congo red and $MgCO_3$, Naphthol yellow S and $MgCO_3$ and Fe_2O_3 and $MgCO_3$.

W. C. Johnson

Thermal

1943. **Thermal capillary analysis with a new multi-sample apparatus.** F. J. Lauer (Inst. Kolloidkunde u. Med.-Biol. Lab., Poppenhausen a.d. Wasserkuppe, Germany). *Fette u. Seifen*, 1955, **57** (10), 789–793.—The apparatus (termed a "Tekameter") consists of a porcelain plate with 20 depressions, in four rows, to take the samples; a framework mounted above the plate carries the paper strips. The ends of the strips can be automatically immersed in the samples while the apparatus is in a thermostat. Errors due to different immersion times and changes of temperature are eliminated. The apparatus is used to determine the capillary rise of various liquids on S. and S. 0860 paper.

E. Hayes

1944. **A rapid method for the determination of moisture in capillary-porous dispersed materials.** V. N. Ershov and N. M. Ershova. *Zh. Tekh. Fiz.*, 1954, **24** (5), 854–858; *Referativnyĭ Zh., Khim.*, 1955, (9), Abstr. No. 16,548.—A method is described for the determination of moisture by the rate of cooling (or heating) of a body in thermal contact with the medium being investigated. Heating curves for quartz sand, chalk, clay, coal and other disperse materials for a wide range of moisture have been obtained by the use of an instrument consisting of two parts: a transmitter (a probe with negligible heat capacity) and an indicator (a galvanometer). It has been established that it is expedient to use as a characteristic the max. rate of heating (or cooling) of the probe, which consists of a cylinder with a working wall of phosphor-bronze plates (2 mm × 20 mm, and 0·15 mm thick), each carrying the hot junction of a copper - constantan thermocouple. The cold junctions of the thermocouples (which are joined in series) lead to the end of the probe. The inner surface of the working wall of the probe is in thermal contact with a heating coil. The probe is immersed in the medium to be tested. The rate of heating of the probe is determined by the rate of change of its thermal e.m.f. It is found that the max. rate of change of thermal e.m.f. of the probe is practically constant in 25 to 30 sec. The construction of an instrument for the rapid determination of moisture content of a porous material by the rate of charge of a condenser is described. The galvanometer scale is calibrated so that the moisture content is read directly from the needle deflection. The error is 0·03 per cent.

C. D. KOPKIN

Electrical

1945. **An electronic computer for the evaluation of results of filter-paper electrophoresis.** M. D. Poulik and L. Pinteric (Univ. Toronto, Ontario, Canada). *Nature*, 1955, **176**, 1226–1227.—An electronic integrator evaluates the area under the whole or a selected part of the distribution curve of a filter-paper electropherogram displayed by a scanning unit and oscilloscope previously described (*Canad. J. Publ. Hlth*, 1954, 163). The quantity of the components corresponding to the separate peaks of the distribution curve can be read directly from a meter as a percentage of the total material. About two minutes are required to evaluate one electropherogram; no treatment of the strip or mathematical operation on data is necessary.

G. A. BASSETT

1946. **Studies on polarography with a mercury-drop control. I. Apparatus and the mercury flow of the dropping electrode.** Yoshiazu Yasumori (Kyoto Univ., Sakyo-ku, Japan). *J. Electrochem. Soc. Japan*, 1955, **23** (11), 578–582.—A device for the synchronisation of two mercury drops for differential polarography was suggested. The dropping of Hg is caused by a mechanical impulse, operated with the aid of a multi-vibrator. The rate of flow of Hg is then lower than that for natural flow. In differential polarography the vibration of current due to the growth of the drop can be elimnated by the use of condensers. The reduction wave of acetaldehyde in the presence of O and Na^+ was examined with the new apparatus. K. SAITO

1947. **The accuracy of quantitative radiometric analyses by the Hevesy - Paneth and the dilution processes.** O. Riedel (Max-Planck Inst. Chem., Mainz, Germany). *Angew. Chem.*, 1955, **67** (21), 643–648.—An investigation was made of the sources of error in radiometric determinations by the Hevesy - Paneth and by the dilution methods, and of the effect of these errors on the results. The errors arise not only from inaccuracies in pipette and weighing measurements but chiefly in the observation of the count rate of the Geiger counters (and the accuracy of the counter apparatus) and in the lack of definition or accuracy of the activity calibration curves. The factors affecting the accuracy of the calibration curves, including the β-ray absorption within the film of active material, are discussed. A method is described for obtaining calibration curves of max. accuracy, and it is shown that there is no practical advantage in using films of active material of $\lesssim 4$ mg per sq. cm in either the construction of the curve or in measuring the activity of the sample. The magnitude of the various errors obtaining in practice is discussed, and the total effect of the final results is calculated by the Gauss method, which shows that the final results are accurate to within 2 to 4 per cent. Control analyses of active thiourea in treated orange skins by the dilution method (with measurement of the emission of the S in the form of $BaSO_4$) give results agreeing to within 2 to 4 per cent. The investigation shows that the methods are sufficiently accurate for analytical purposes.

H. L. WHITEHEAD

1948. **A self-pumping dropping-mercury electrode for use with stirred solutions.** D. M. Miller (Science Service Lab., London, Ontario, Canada). *Canad. J. Chem.*, 1955, **33** (6), 1184–1187.—A modified Lingane - Laitinen H cell (*Ind. Eng. Chem., Anal. Ed.*, 1939, **11**, 504) is described for polarography with stirred soln. The dropping-mercury electode is inserted in a side arm, which is connected to the H cell by two capillaries. Mercury drops fall into the lower capillary and draw fresh soln. from the main bulk contained in the H cell through the upper capillary, so that the soln. round the electrode is completely changed every two or three drops. With drop times of 4 sec. or more, the effect of the circulation of soln. on the polarogram is negligible.

H. F. W. KIRKPATRICK

1949. **An oscilloscopic polarograph.** G. F. Atkinson and W. A. E. McBryde (Univ. Toronto, Ontario, Canada). *Canad. J. Chem.*, 1955, **33** (7), 1272–1275.—Details are given of a circuit for an oscilloscopic polarograph incorporating phase-shifting components to produce elliptical Lissajous patterns, and an electronic switch in the oscilloscope's vertical input to provide a reference signal in addition to that observed for the polarographic cell. In operation, the two signals are matched, superimposed, and the point of no deviation is observed to give $E_{\frac{1}{4}}$.

H. F. W. KIRKPATRICK

1950. **Use of oscillographic polarography in quantitative analysis. IV. Apparatus for measuring the depths of cut-ins on oscillographic curves.** R. Kalvoda (Polarographic Inst., Czech. Acad. Sci., Prague, Czechoslovakia). *Chem. Listy*, 1955, **49** (5), 759–762.—An arrangement for the simultaneous representation of the oscillographic curve and a horizontal axis is described. The depth of the cut-ins on the curves of the functions $dV/dt = f(V)$ and $dV/dt = f(t)$ can be determined by measuring the distance between the apex of the cut-in and the projected axis. The depth being a

function of the concn. of the depolariser, the latter can be readily determined by means of a calibration curve. G. GLASER

1951. **Measurement of differential capacity and the resistance of the solution with an oscillographic polarograph.** Eiji Niki (Inst. of Ind. Sci., Tokyo Univ., Nishichiba). *J. Electrochem. Soc. Japan*, 1955, **23** (10), 526–530.—The differential double-layer capacity of the dropping-mercury electrode and the resistance of the soln. were measured with various supporting electrolytes. The parallel equilibrium capacity measured at a low frequency agrees well with the differential double-layer capacity. The resistance of the soln. is calculated from the reciprocal of the equiv. conductance at a high frequency. Examples are shown with Zn and Cd ions in NH_4Cl - aq. NH_3 and NaOH soln.
K. SAITO

1952. **Effect of highly radioactive solutions on glass pH electrodes.** J. H. Pannell (Oak Ridge Nat. Lab., Tennessee, U.S.A.). *Nucleonics*, 1955, **13** (8), 48–49.—Two Beckman general-purpose glass electrodes, type 1190–80, and one, type 290–1, with a small tip, were immersed in acid solution containing $\simeq 0{\cdot}4$ curie of radioactivity per ml due to ^{144}Ce - ^{144}Pr. Although the glass darkened after 1 hr. the electrode potentials had not changed markedly after three days' exposure. E. N. JENKINS

1953. **New electrode for titration in non-aqueous medium.** J. Minczewski and Z. Łada (Zaklad Anal. Inst. Chem., Ogólnej, Warsaw). *Roczn. Chem.*, 1955, **29**, 919–920.—Difficulties of potentiometric titrations in anhydrous organic solvents (slow stabilisation and irregularities of potential, caused by the surface contact between the S.C.E. and non-aq. solvent) are overcome by using an Ag - AgCl electrode mounted in a glass tube containing (*a*) a saturated solution of KCl in glacial acetic acid (for titrations in acetic acid medium) or (*b*) a 10 per cent. methanolic solution of LiCl (for titrations with Na or K methoxide). The contact between electrode and test solution is established by means of a liquid - liquid junction through a ground joint at the bottom of the glass tube.
S.C.I. ABSTR.

1954. **A new micro-coulometer.** E. Schwarz-Bergkampf and R. Reichert (Montanistischen Hochschule, Leoben, Austria). *Mikrochim. Acta*, 1955, (5–6), 1031–1036.—A detonating-gas coulometer of great accuracy, based on the principle of pressure measurement at constant volume, is described. C. A. SLATER

1955. **Automatic gas analyser for hydrogen sulphide.** B. M. Maslennikow and F. A. Kavickaja. *Chem. Tech., Berlin*, 1955, **7** (11), 682–683.—A detailed description is given of an automatic photo-electric analyser for detecting small amounts of H_2S in air or other gases. It is particularly designed to give warning when the H_2S content of the gas is above an unwanted limit. The stream of gas to be analysed is directed on to a travelling strip of detector paper (which is impregnated with Pb acetate) at a point near that at which a beam from a standard illuminating lamp traverses the paper to fall onto a photo-electric cell. The detector paper becomes opaque to a degree corresponding to the H_2S content of the gas stream, and thereby varies the current set up by the cell. These variations are relayed and valve-amplified, and are made to operate a warning bell when the opacity rises to an extent corresponding to an unwanted high concn. of H_2S. H. L. WHITEHEAD

1956. **Rectified radio-frequency method (R.Rf.) of zone location for paper chromatography.** G. G. Blake (Univ. Sydney, N.S.W., Australia). *Anal. Chim. Acta*, 1955, **13** (6), 562–565.—A brief review of the applications of radio-frequency oscillations to chemical analysis is followed by a description of a zone locator for paper chromatography. This comprises a new variation of the condenser principle. The electrical circuit uses a transistor for rectification and amplification, and is of simple construction.
W. C. JOHNSON

1957. **Scintillation counter for the assay of radioactive gases.** D. R. Stranks (Univ. Leeds, England). *J. Sci. Instrum.*, 1956, **33** (1), 1–4.—Weak β-emitting nuclides (^{14}C) in the gas phase can be estimated with a reproducibility of 0·5 per cent. in a glass chamber having a plastic scintillator as one wall. The counter assembly can be adapted to the assay of both liquid and solid samples.
G. SKIRROW

1958. **Liquid scintillation counter for ^{14}C employing automatic sample alternation.** A. J. Weinberger, J. B. Davidson and G. A. Ropp (Oak Ridge Nat. Lab., Tenn., U.S.A.). *Anal. Chem.*, 1956, **28** (1), 110–112.—An instrument is described in which a single refrigerated IP21 detector and one amplifier are used to count three specimens alternately by a mechanical device. By making one a standard, one the background and the third the sample, the effects of drift are eliminated. Performances over extended periods are described and the average deviation from the mean and from the calculated values is $\simeq \pm 0{\cdot}5$ per cent. P. T. BEALE

ABBREVIATIONS

Certain abbreviations in everyday use are not included in the following list. When any doubt might arise from the use in the text of an abbreviation or symbol the word is printed in full.

alternating current	a.c.
ampere	amp.
Ångstrom unit	Å
anhydrous	anhyd.
approximate, -ly	approx.
aqueous	aq.
atmospher, -e, -ic	atm.
boiling-point	b.p.
British thermal unit	B.Th.U.
calorie (large)	kg-cal.
calorie (small)	g-cal.
centimetre	cm
coefficient	coeff.
concentrated	conc.
concentration	concn.
critical	crit.
crystalline	cryst.
crystallised	cryst.
cubic	cu.
current density	c.d.
cycles per second	c.p.s.
decompos -ing, -ition	(decomp.)
density	ρ
density, relative	d or wt. per ml
derivative	deriv.
dilute	dil.
direct current	d.c.
distilled	dist.
electromotive force	e.m.f.
electron-volt	eV
equivalent	equiv.
experiment	expt.
foot, feet	ft.
gram	g
gram-molecule	mole
half-wave potential	$E_{\frac{1}{2}}$
horse-power	h.p.
hour	hr.
hydrogen ion concentration	$[H^{\cdot}]$
hydrogen ion exponent	pH
inch	in.
infra-red	i.r.
insoluble	insol.
kilogram	kg
kilovolt	kV
kilowatt	kW
maxim -um, -a	max.
melting-point	m.p.
microcurie	μC
microgram	μg
microlitre	μl
micron	μ
milliampere	mA
millicurie	mC
milligram	mg
millilitre	ml
millimetre	mm
millimicron	mμ
millivolt	mV
minimum	min.
minute (time)	min.
molar (concentration)	M
molecul -e, -ar	mol.
normal (concentration)	N
number	no.
observed	(obs.)
ounce	oz
part	pt.
patent	pat.
parts per million	p.p.m.
per cent. wt. in wt.	per cent. w/w
per cent. wt. in vol.	per cent. w/v
per cent. vol. in vol.	per cent. v/v
potential difference	p.d.
pound	lb
precipitate	ppt.
precipitated	pptd.
precipitating	pptg.
precipitation	pptn.
preparation	prep.
qualitative, -ly	qual.
quantitative, -ly	quant.
recrystallised	recryst.
refractive index	n^t_λ
relative humidity	R.H.
revolutions per minute	r.p.m.
saponification value	sap. val.
saturated calomel electrode	S.C.E.
second (time)	sec.
soluble	sol.
solution	soln.
specific gravity	sp. gr.
specific rotation	$[\alpha]^t_\lambda$
square centimetre	sq. cm
standard temperature and pressure	s.t.p.
temperature	temp.
ultra-violet	u.v.
vapour density	v.d.
vapour pressure	v.p.
volt	V
volume	vol.
watt	W
wavelength	λ
weight	wt.

In addition the following symbols are used—

greater than	$>$	less than	$<$
not greater than	$\ngtr$	not less than	$\nless$
is proportional to	$\propto$	of the order of, approximately	$\simeq$

The principal Pharmacopoeias are denoted by B.P., U.S.P., or D.A.B., together with the identifying numeral.

Radicles are represented by the usual symbols; positive ions have superscript dots and negative ions superscript dashes, *e.g.*, $Cu^{\cdot\cdot}$, $Al^{\cdot\cdot\cdot}$, Cl', SO_4''. Metals that exist in more than one valency state are represented by their symbols with appropriate superscript roman numerals, *e.g.*, ferric iron becomes Fe^{III} and cuprous copper Cu^{I}.

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